

APPENDIX M
PROTOCOL
FOR COLLECTION OF COMPOSITE SOIL SAMPLES

PROTOCOL FOR COLLECTION OF COMPOSITE SOIL SAMPLES

1.0 Introduction

Soil samples will be collected with a clean 1 1/8-inch internal diameter (I.D.) soil recovery probe inserted into the ground to a depth of approximately 2 inches. The soil recovery probe consists of a 6-inch stainless steel core sampler, replaceable 1-in I.D. butyrate plastic inserts, a cross-bar handle, and a hammer attachment (Figure 1). A composite sample consisting of three soil cores will be collected at each location specified in the QAPjP. The top 1/2 inch section of the soil cores will be composited at the site by the field team. Some dwellings may be surrounded only by concrete or blacktop. This situation would preclude soil sampling.

In this study, soil core samples are scheduled for collection from study dwellings at the following campaigns: initial sampling campaign (i.e enrollment campaign for control homes and pre-intervention campaign for R&M homes), at immediate post-R&M for R&M dwellings, and at the 6 month and 18 month campaigns. In all homes, sampling will include a drip line sample near the property foundation and a Field Blank. The 6 month campaign will also include a sample from the property boundary. Ten percent of the homes will also include a field duplicate sample.

2.0 Sampling Equipment and Supplies

1 1/8-in diameter, stainless-steel, soil-recovery probe with cross-bar handle, 6-in length (Arts Manufacturing and Supply, American Falls, Idaho).

AMS hammer attachment for hard, dry, or lightly frozen soils.

1-inch I.D. butyrate plastic liner inserts.

1-in diameter plungers with and without adjustable stop.

Plastic straight edge (ruler).

Clamp for holding liner (optional).

Vinyl gloves (powderless).

Hard plastic storage containers for sampling kits/supplies.

1-gallon ziplock plastic bags.

Plastic trash bags.

Soil Sampling Kits with barcode labels (at least 8 identical labels per sample with a unique sample number). One kit per sample.

Soil Core Collection forms.

Field Sample Traceability forms.

Knee Pads.

Clip board.

Measuring tape.

Crescent wrenches (2 per team) for disassembly of soil sampling probe.

Wash bottle.

95% Ethanol

Wash-a-Bye Baby Wipes

3.0 Soil Sampling Kits

The Sample Custodian will prepare soil sampling kits for each sampling site. Each soil sampling kit will consist of a 1-inch I.D. butyrate plastic liner insert, two 1-gallon ziplock plastic bags and at least 8 identical adhesive barcode labels. The plastic liner will come doubled sealed in the two ziplock bags. The inner bag will be prelabelled with one of the barcode labels. The remainder of the adhesive labels will be contained in the outer plastic bag. Optional: To distinguish the top of the soil core from the bottom, the outside of the plastic liners can be marked with an arrow pointing to the bottom end of the liner using a permanent marking pen.

To avoid contamination of the sampling materials, the kits should not be opened until they are needed in the field.

4.0 Soil Sampling Protocol

The following protocol will be used for collecting the soil samples:

Don a pair of clean powderless vinyl gloves.

Remove a corresponding barcode label from the outer bag of an unused sampling kit and affix it to the Soil Core Collection form in the appropriate space for SAMPLE ID. This is done by the team leader.

Disassemble a clean soil recovery probe (unscrew the soil probe section from the coupling).

Open the inner prelabelled ziplock bag and remove the plastic liner from the soil sampling kit.

Remove the protective end caps from the plastic liner (the end caps are optional when the liner is sealed inside a ziplock bag).

Insert the plastic liner into the probe (arrow pointing down toward the tip).

Reassemble the probe and attach the cross-bar handle or hammer attachment.

Push the soil recovery probe into the soil to a depth of approximately 2 inches.

Twist and snap the coring tool to one side and remove the core sample.

Disassemble the probe and remove the plastic liner containing the core sample.

Insert a clean 1-in diameter plunger into the top end of the liner.

Orient the liner with the arrow pointing up.

Push out all but 1/2 inch of the core from the liner with the plunger.

Note: A plunger equipped with an adjustable stop is recommended. The stop can be adjusted to prevent the plunger from advancing beyond 1/2 from the end of the liner. If necessary, the liner can be secured in a clamp during this procedure. The use of a clamp is recommended when the sampling is performed by one person.

Scrape the top of the liner with a clean straight edge to level off soil that was pushed out of the liner. Discard the soil pushed out of the liner.

Note: The soil can also be leveled off with a gloved finger. This may be a faster method.

With a clean plunger (without stop), push the remaining 1/2 inch section of the core sample into the prelabelled ziplock bag.

Note: A second plunger without an adjustable stop is recommended for pushing the remaining section of the core out of the liner.

Reinsert the plastic liner into the soil recovery probe and reassemble the unit.

Collect the remaining cores of the composite sample as per the QAPjP using the same method as described above. The three cores that constitute the composite sample are placed into the same ziplock plastic bag.

Return the inner plastic bag that now contains the composite sample to the original outer ziplock plastic bag that contains the remaining barcode labels and seal.

After each composite sample is collected, discard the plastic liner in the trash bag.

Remove another corresponding barcode label from the soil sampling kit and affix it to the Field Traceability Form.

Wipe down the recovery probe, plungers, and straight edge with Wash-a-Bye baby disposable wipes. Discard the wipes in the trash bag. If conditions are cold, dampen each wipe prior to use with 95 percent Ethanol

to help prevent water in the wipes from forming ice on the equipment.

Remove the vinyl gloves and discard in the trash bag.

Record site location, sampling location, date, etc. on the Soil Core Collection Form. (This function will be performed by the team leader).

Transport samples to the KKI Trace Metals Laboratory.

4.1 Alternative Sampling Procedures for Hard, Dry, or Frozen Soils

The following is an alternate soil sampling protocol that will be used for soils that are hard, dry, or frozen:

Remove the cross-bar handle from the soil recovery probe.

Attach the AMS hammer to the probe.

Note: Some field team members may prefer to use the hammer attachment for all soil samples.

Grip the hammer attachment firmly and drive the probe into the ground to a depth of approximately 2 inches using an up and down motion.

If conditions do not allow for full penetration to 2 inches, make every effort to penetrate to a depth of at least 1/2 inches. If the penetration is less than 2 inches, note the deviation from the protocol on the sampling data form.

5.0 Preparation of Field Blanks for the Soil Sampling Procedure

One soil core Field Blank (FB) will be processed at each dwelling during each campaign in which soil core samples are collected. A Field Blank (FB) will consist of a core liner that is loaded into the soil recovery probe and handled in the same manner as the regular samples except no soil sample is collected. The following procedure will be used:

Field Blank Procedures:

Don a pair of clean powderless vinyl gloves.

Remove a corresponding barcode label from the outer bag of an unused sampling kit and affix it to the Soil Core Collection form in the appropriate space for Field Blank (FB). This is done by the team leader.

Disassemble a clean soil recovery probe that has been cleaned (unscrew the soil probe section from the coupling).

Open the inner prelabelled ziplock bag and remove the plastic liner from the soil sampling kit.

Remove the protective end caps from the plastic liner (the end caps are optional when the liner is sealed inside a ziplock bag).

Insert the plastic liner into the probe (arrow pointing down toward the tip).

Reassemble the probe.

Disassemble the probe and remove the plastic liner without collecting a sample.

Insert the clean plungers into the liner by the same method that is normally used to extract the soil core from the liner.

Scrape the top of the liner with a clean straight edge or with gloved finger as done during the sampling procedure.

Replace the end caps on the liner and place the capped liner inside the original prelabeled ziplock bag.

Return the inner plastic bag that now contains the Field Blank to the original outer ziplock plastic bag that contains the remaining barcode labels and seal.

Remove another corresponding barcode label from the soil sampling kit and affix it to the Field Traceability Form. (This function will be performed by the team leader).

Remove the vinyl gloves and discard in the trash bag.

Transport the Field Blank with the other soil samples to the KKI Trace Metals Laboratory.

6.0 Collection of Field Duplicates

In ten percent of the homes, a Field Duplicate (FD) sample will be collected using the same protocol as described above after the first sample and the Field Blank have been collected. The Field Duplicate sample will be collected as close as possible to the locations sampled for the routine soil composite sample. The Soil Core Collection Form indicates the dwelling IDs of the homes where field duplicates are to be collected.

In order to link the sampling data of the routine sample and its Field Duplicate sample, the I.D. number of each of the samples must be indicated on the Soil Core Collection form of the sample collected adjacent to it. This will create the bridge between the two data sets. These co-located samples will be handled and transported to the KKI Trace metals Laboratory with the regular soil samples.

7.0 Contamination Avoidance

The following work practices will be instituted to prevent contamination of the sample and cross contamination between each composite soil sample collected:

Soil core liners will be acid rinsed prior to going out to the field. (This is done by the Sample Preparation Technician prior to assembly of soil sampling kits).

Soil samples should not be collected until all dust samples are collected within the house.

Clean vinyl gloves (powderless) will be donned prior to collecting each soil/dirt sample and will be disposed of after the collection of each composite sample.

The soil recovery probe, plungers, and straight edge will be cleaned with wet disposable wipes between each composite sample (i.e. collect all 3 cores that constitute the composite sample and then clean the sampling materials before collecting the next sample).

8.0 Deviations from Field Sampling Protocols

Every attempt shall be made to follow this soil core collection protocol. Deviations from the sampling protocols may compromise the data quality and completeness objectives of the project. Deviations from the protocols will generally fall into two categories: inadvertent deviations (procedural errors); and deliberate deviations (modifications to the protocol in response to unusual conditions encountered in the field).

In the case of any deviation from the protocol, the sampling team shall fully document the deviation on the Soil Core Collection Form in the comment space provided and immediately notify the Outreach Coordinator, the Project Manager, and the QC Officer. In the case of inadvertent deviations from protocol, corrective action(s) shall be taken to ensure that the situation is not repeated. If possible, samples affected by the inadvertent deviation (e.g. sample is spilled in the field) should be recollected in accordance with the specified protocol prior to leaving the site. Note that for each field voided sample, a corresponding barcode label should be placed on the Field Traceability Form in one of the spaces reserved for field voided sample barcode labels.

The Outreach Coordinator and QC Officer shall be notified by the sampling team when field conditions found at the sampling site do not allow full compliance with the protocol or when the protocol does not appear to apply to the situation. The condition/ situation shall be fully documented in a field notebook. The Outreach Coordinator will in turn notify the Project Manager.

Any permanent changes/modifications in this formal sampling protocol must be approved in advance in writing. In the event that permanent changes/modifications are made, field teams will receive copies of the modified written protocol and training as needed.

APPENDIX N
PROTOCOL
FOR COLLECTION OF DRINKING WATER SAMPLES

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PROTOCOL FOR COLLECTION OF DRINKING WATER SAMPLES

1.0 Introduction

Samples will be fixed-time (2 hour) stagnation samples collected from the kitchen sink faucet if possible. First flush samples will not be collected due to concerns for family cooperation and worker safety during early morning sampling necessitated by first flush sampling. The fixed-time stagnation samples are collected by first running the water for at least 2 minutes to clear the line of water standing in the pipe. The sample is collected, two hours after clearing the line, as the first stream of water from the faucet. This type of sample would represent water standing in the house plumbing for a fixed amount of time. Lead-containing solder is the most common source of lead in drinking water. Members of the household will be instructed to refrain from using any water from the selected faucet until all water samples are collected.

In this study, drinking water samples are scheduled for collection from study dwellings at the following campaigns: initial sampling campaign (i.e enrollment campaign for control homes and pre-intervention campaign for R&M homes), and at the 6 month and 18 month campaigns. In all homes, sampling will include the fixed-time stagnation sample and a field blank. Ten percent of the homes will also include a field duplicate sample.

2.0 Sampling Equipment and Supplies

Container for kits which keeps the bottles upright.

Nalgene wide-mouth, high-density plastic bottles (500 mL) pre-loaded with acid.

Barcode labels (a strip of at least 8 identical labels per sample with a unique sample number).

1-gallon ziplock plastic bags.

Disposable vinyl gloves (powderless).

Water Collection and Field Sample Traceability Forms.

Post-It Notes or sign (to remind family not to use faucet)

Note: Field teams should have available in the study vans spill cleanup supplies and a copy of the spill cleanup instructions.

3.0 Water Sampling Kits

A water sampling kit will consist of a pre-cleaned wide-mouth Nalgene bottle (500 mL) pre-loaded with acid, and a strip of at least 8 identical barcode labels packaged together in two 1-gallon ziplock bags (see below). At least four kits should be taken to the field for each dwelling. This will allow for the collection of the fix-time stagnation sample, the field blank, the field duplicate (in 10% of the homes) and leave at least one kit as a backup kit. Important: Sample kits taken to the field and not used (e.g. the spare kit used for backup purposes) should be included for use in the next day's sampling or otherwise as soon as possible. This will avoid having bottles pre-loaded with acid remaining unused for extended periods of time.

The Sample Preparation technician will add 20 mL of 25% (v/v) nitric acid solution to acid-cleaned Nalgene sampling bottles and prepare the sampling kits as needed. The acid will be added to the bottles as close as possible to time of sampling. One barcode label (1 of at least 8 identical labels) will be affixed to the outside surface of the Nalgene bottle. The bottle will then be placed into a 1-gallon ziplock bag and sealed. This bag will be placed into a second plastic bag containing the remainder of the corresponding barcode labels.

4.0 Fix-time Stagnation Drinking Water Sampling Procedure

Drinking water samples will be collected as close as possible to two hours following the flush of the pipes. It is important to record on the Water Sample Collection Form both the time the flush of the pipes is completed and the time of sampling. This will enable us to calculate the amount of time that the water sample was in contact with the house plumbing. The following procedure will be used to collect the sample:

Upon entry into the housing unit, check that the kitchen faucet is available for sampling and that it does not have a steady drip. If needed, check a bathroom sink faucet as a backup sampling site. Run the water from the selected faucet for two minutes to clear the line and then close the tap. If sampling can only be done from a dripping faucet, then note this on the appropriate comment section of the data collection form.

Record on the Water Sample Collection Form the time that the line was cleared. (See heading labeled "TIME SYSTEM FLUSH COMPLETED.")

Instruct the occupants of the house to refrain from using any water from the selected faucet between the time the line is flushed and the time of sampling. If possible, post a reminder note or sign on or near the selected faucet.

Return to the faucet selected for water collection as close as possible to 2 hours after the line was flushed.

Don a pair of clean vinyl gloves.

Remove a corresponding barcode label from a water sampling kit and affix it to the Water Sample Collection Form in the appropriate space for SAMPLE ID.

Remove the Nalgene bottle from one of the water sampling kits (inner bag) and remove the lid.

Place the opened bottle under the selected faucet and slowly open the cold water valve.

Collect the sample by slowly filling the plastic container to the neck with water from the selected faucet. When the bottle is filled, close the water valve and replace the lid on the bottle.

Return the bottle to the original ziplock bag and seal.

Record the time that the sample was collected on the Water Sample Collection Form.

Remove another corresponding barcode label from the water sampling kit and affix it to the Field Traceability Form.

5.0 Preparation of Field Blanks

One drinking water Field Blank (FB) will be processed at each dwelling during each campaign in which drinking water samples are collected. A field blank will consist of a Nalgene collection bottle that will be handled in the field following the same protocol as described above in section 4.0 except that no water will be collected. The Field Blank will be prepared in accordance with the procedures listed below.

Field Blank Procedures:

Don a pair of clean vinyl gloves.

Remove one corresponding barcode label from a water sampling kit and affix it to the Water Sample Collection Form in the space reserved for the Field Blank (FB) barcode label.

Remove the Nalgene bottle from one of the water sampling kits (inner bag) and remove the lid.

Place the opened bottle under the selected faucet and then close the lid without collecting a sample.

Return the bottle to the original ziplock bag and seal.

Remove another corresponding barcode label from the water sampling kit and affix it to the Field Traceability Form.

Repackage the bottle in its original ziplock bag. The Field Blank will be packaged and transported to the Kennedy Krieger Institute Trace Metal Laboratory (KKI-TML) with the other water samples.

6.0 Collection of a Sequential Water Sample (Field Duplicate)

In ten percent of the homes, a second drinking water sample will be collected from the same faucet immediately after collecting the drinking water sample and the Field Blank using the same procedures as described below. This sample is referred to as the Field Duplicate (FD). The Water Sample Collection Form indicates the dwelling IDs of the homes where field duplicates are to be collected.

Don a pair of clean vinyl gloves.

Remove a corresponding barcode label from a water sampling kit and affix it to the Water Sample Collection Form in the space reserved for the Field Duplicate barcode label. This barcode label provides a link between the Field Duplicate and the first drinking water sample.

Remove the Nalgene bottle from one of the water sampling kits (inner bag) and remove the lid.

Place the opened bottle under the selected faucet and slowly open the cold water valve.

Collect the sample by slowly filling the plastic container to the neck with water from the selected faucet. When the bottle is filled, close the water valve and replace the lid on the bottle.

Return the bottle to the original ziplock bag and seal.

Remove another corresponding barcode label from the water sampling kit and affix it to the Field Traceability Form.

This sample will be packaged and transported to the KKI-TML along with the other water samples.

7.0 Contamination Avoidance

The following work practices will be instituted to prevent contamination of the sample and to prevent cross contamination between sampling sites:

Prewashing Water Collection Bottles with acid in the laboratory. (Done by Sample Preparation Technician).

Donning of shoe coverings by Field Teams prior to entering each dwelling to prevent cross contamination between sampling sites.

Not opening the water sampling kits until needed in the field.

Donning clean disposable vinyl gloves prior to collecting each water sample.

Placing the sample bottle cap in or on the 1-qt ziplock bag after uncapping the bottle.

8.0 Procedures for Cleanup of Spills in the Field

See the attached instructions for cleaning up acid and water sample spills in the field.

9.0 Deviations from the Water Collection Protocol

Every attempt shall be made to follow this water collection protocol. Deviations from the sampling protocols may compromise the data quality and completeness objectives of the project. Deviations from the protocols will generally fall into two categories: inadvertent deviations (procedural errors); and deliberate deviations (modifications to the protocol in response to unusual conditions encountered in the field).

In the case of any deviation from the protocol, the sampling team shall fully document the deviation on the Water Sample Collection Form in the comment space provided and immediately notify the Outreach Coordinator, the Project Manager, and the QC Officer. In the case of inadvertent deviations from protocol, corrective action(s) shall be taken to ensure that the situation is not repeated. If possible, samples affected by the inadvertent deviation (e.g. sample is spilled in the field) should be recollected in accordance with the specified protocol prior to leaving the site. Note that for each field voided sample, a corresponding barcode label should be placed on the Field Traceability Form in one of the spaces reserved for field voided sample barcode labels.

The Outreach Coordinator and QC Officer shall be notified by the sampling team when field conditions found at the sampling site do not allow full compliance with the protocol or when the protocol does not appear to apply to the situation. The condition/ situation shall be fully documented in a field notebook. The Outreach Coordinator will in turn notify the Project Manager.

Any permanent changes/modifications in this formal sampling protocol must be approved in advance in writing. In the event that permanent changes/modifications are made, field teams will receive copies of the modified written protocol and training as needed.



ACID NEUTRALIZER AND CHEMICAL BURN SOLUTIONS

DO NOT APPLY THE ACID NEUTRALIZER TO SKIN; USE ONLY NEUTRA-SOL FOR SKIN APPLICATIONS.

In order to ensure safety in the field from possible spills of the water sampling containers containing 25% HNO_3 , the following procedure should be followed.

1. Wear protective gloves when handling sampling vessels which contain acid.
2. If a spillage occurs use the Acid Neutralizer which is provided for each team and should be taken each day as part of the sampling kits.
3. Simply spray the solution on the acid until the color turns from yellow (which indicates acid) to purple which indicates neutralization.
4. The acid neutralizer can not be to applied to the skin. Any acid spilled on the skin should be immediately treated with Neutra-Sol, a sterile phosphate buffer which is included in the preparation of the field kits.
5. After collection of the water sample the acidity of the sample will be less than 0.1% and will not constitute a hazard under normal circumstances.



APPENDIX O

SAMPLE COLLECTION FORMS

1. Dwelling Unit Cover Sheet
2. Dust-Vacuum Collection Forms for Floors, Air Ducts, Upholstery, Windows and Field QC
3. Wipe-Dust Collection Forms for Floors, Air Ducts, Windows, Field QC, and Side-by-Side Wipe/Vacuum
4. Soil Collection Form
5. Drinking Water Collection Form
6. Blood-Lead Collection Form
7. Environmental Sampling Checklist
8. Example of Diagram with Sampling Plan



Kennedy Krieger Institute Repair and Maintenance Study
DWELLING UNIT COVERSHEET

Dwelling ID Round

Address: _____

Date: _____ Initials: _____

Child Name _____ DOB ____/____/____ Child ID

Child Name _____ DOB ____/____/____ Child ID

Child Name _____ DOB ____/____/____ Child ID

Child Name _____ DOB ____/____/____ Child ID

Child Name _____ DOB ____/____/____ Child ID

Checklist:

| | | | |
|----------------|----------------|----------------|----------------|
| Section A ____ | Section D ____ | Section E ____ | Section F ____ |
| Section B ____ | Section E ____ | Section F ____ | Section E ____ |
| Section C ____ | Section F ____ | Section E ____ | Section F ____ |

Clinic Appointment(s): _____

Supervisor: _____ Date: _____



**Kennedy Krieger Institute Repair and Maintenance Study
DUST/VACUUM COLLECTION FORM FOR FLOORS/AIR DUCTS/UPHOLSTERY**

DWELLING ID - ROUND

(Round: IN PI 02 06 12 18 24)

| | | | | | |
|----------------------|----------------------|----------------------|---|----------------------|----------------------|
| <input type="text"/> | <input type="text"/> | <input type="text"/> | - | <input type="text"/> | <input type="text"/> |
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DATE

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| m | m | | d | d | | y | y |

SAMPLER (Initials)

| | |
|----------------------|----------------------|
| <input type="text"/> | <input type="text"/> |
|----------------------|----------------------|

RECORDER (Initials)

| | |
|----------------------|----------------------|
| <input type="text"/> | <input type="text"/> |
|----------------------|----------------------|

LEVEL _____

SURFACE TYPE

F = Floor I = Front Interior Entrance E = Front Exterior Entrance U = Upholstery
A = Air Duct B = Rear Interior Entrance R = Rear Exterior Entrance

| |
|----------------------|
| <input type="text"/> |
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SAMPLE TYPE

W = Composite of rooms with windows I = Individual P = Play Area
N = Composite of rooms with no windows S = Vac/Wipe Pair

| |
|----------------------|
| <input type="text"/> |
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(Enter Room Letter to be Sampled)

| | | | | | | | | | | |
|--|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|
| Room Letter | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| Surface Material⁽¹⁾ | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| Surface Condition⁽²⁾ | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |

Place
Bar Code
Here

KEY: (1) W = Sealed Wood L = Smooth Linoleum/Tile F = Fiber/Carpet B = Marble
 U = Unsealed Wood T = Textured Linoleum/Tile C = Concrete O = Other
 (2) S = Smooth R = Rough(intact) D = Rough(deteriorated) N = Not Applicable(fiber/carpet)

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other

DESCRIBE:

LEVEL _____

SURFACE TYPE

(Enter Code: F, I, E, U, A, B, or R)

SAMPLE TYPE

(Enter Code: W, I, P, N, or S)

(Enter Room Letter to be Sampled)

| | | | | | | | | | | |
|--|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|
| Room Letter | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| Surface Material⁽¹⁾ | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |
| Surface Condition⁽²⁾ | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> |

Place
Bar Code
Here

KEY: (1) W, L, F, B, U, T, C, or O
(2) S, R, D, or N

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other

DESCRIBE:

Supervisor: _____ **Date:** _____



**Kennedy Krieger Institute Repair and Maintenance Study
DUST/VACUUM COLLECTION FORM FOR WINDOWS**

DWELLING ID - ROUND

(Round: IN PI 02 06 12 18 24)

DATE

SAMPLER (Initials)

RECORDER (Initials)

LEVEL _____

SURFACE TYPE S = Window Sill W = Window Well

SAMPLE TYPE C = Composite R = Right Half Composite L = Left Half Composite
 S = Vac/Wipe Pair I = Individual P = Play Window

(Enter Window Number to be Sampled)

| | | | | | | | | | | |
|--|--|--|--|--|--|--|--|--|--|----|
| Window No.: | | | | | | | | | | 24 |
| Surface Material⁽¹⁾ | | | | | | | | | | 40 |
| Surface Condition⁽²⁾ | | | | | | | | | | 48 |
| Paint Chips⁽³⁾ | | | | | | | | | | 56 |

KEY: (1) V = Vinyl M = Metal W = Scaled Wood U = Unscaled Wood
 (2) S = Smooth R = Rough(intact) D = Rough(deteriorated)
 (3) 0 = No 1 = Yes

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other

DESCRIBE:

LEVEL _____

SURFACE TYPE (Enter Code: S or W)

SAMPLE TYPE (Enter Code: C, R, L, S, I, or P)

(Enter Window Number to be Sampled)

| | | | | | | | | | | |
|--|--|--|--|--|--|--|--|--|--|----|
| Window No.: | | | | | | | | | | 24 |
| Surface Material⁽¹⁾ | | | | | | | | | | 40 |
| Surface Condition⁽²⁾ | | | | | | | | | | 48 |
| Paint Chips⁽³⁾ | | | | | | | | | | 56 |

KEY: (1) (Enter Code: V, M, W, or U)
 (2) (Enter Code: S, R, or D)
 (3) (Enter Code: 0 or 1)

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other

DESCRIBE:

Supervisor: _____ **Date:** _____



Kennedy Krieger Institute Repair and Maintenance Study
DUST/VACUUM COLLECTION FORM FOR FIELD QC:
Field Blanks and Field Duplicates

DWELLING ID - ROUND

(Round: IN PI 02 06 12 18 24)

DATE

SAMPLER (Initials)

RECORDER (Initials)

FIELD BLANK SAMPLE

(One per dwelling)

Place FB
Bar Code
Here

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other
DESCRIBE:

FIELD DUPLICATE SAMPLE

(If the last digit of the dwelling ID is a "2" then
collect a field duplicate, otherwise leave blank.)

Place FD
Bar Code
Here

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other
DESCRIBE:

FIELD DUPLICATE LINK

(Put Bar Code of Dust Vacuum Sample paired to Field Duplicate)

Place Bar Code
of DV Sample
Paired with FD

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other
DESCRIBE:

Supervisor: _____ Date: _____

11/16/92



**Kennedy Krieger Institute Repair and Maintenance Study
WIPE DUST COLLECTION FORM FOR FLOORS/AIR DUCTS**

DWELLING ID - ROUND

(Round: IN PI 02 06 12 18 24)

DATE

SAMPLER (Initials)

RECORDER (Initials)

SURFACE TYPE

F = Floor

A = Air Duct

E = Exterior Entrance

I = Front Interior Entrance

R = Rear Interior Entrance

WIPE SAMPLE TYPE

I = Individual

P = Play Area

(Enter Room Letter to be Sampled)

| | | |
|--|--|----|
| Room Letter | | 24 |
| Surface Material⁽¹⁾ | | 25 |
| Surface Condition⁽²⁾ | | 26 |

KEY: (1) W = Sealed Wood

L = Smooth Linoleum/Tile

F = Fiber/Carpet

B = Marble

U = Unsealed Wood

T = Textured Linoleum/Tile

C = Concrete

O = Other

(2) S = Smooth

R = Rough(intact)

D = Rough(deteriorated)

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other

DESCRIBE:

SURFACE TYPE

(Enter Code: F, A, E, I, or R)

WIPE SAMPLE TYPE

(Enter Code: I, or P)

(Enter Room Letter to be Sampled)

| | | |
|--|--|----|
| Room Letter | | 24 |
| Surface Material⁽¹⁾ | | 25 |
| Surface Condition⁽²⁾ | | 26 |

KEY: (1) W, L, F, B, U, T, C, or O

(2) S, R, or D

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other

DESCRIBE:

Supervisor: _____ **Date:** _____



**Kennedy Krieger Institute Repair and Maintenance Study
WIPE DUST COLLECTION FORM FOR WINDOWS**

DWELLING ID - ROUND

(Round: IN PI 02 06 12 18 24)

DATE

SAMPLER (Initials)

RECORDER (Initials)

SURFACE TYPE

S = Window Sill W = Window Well

WIPE SAMPLE TYPE

E = Entire Surface R = Right Half L = Left Half P = Play Window

(Enter Window Number to be Sampled)

| | |
|----------------------------------|----|
| Window No.: | 24 |
| Surface Material ⁽¹⁾ | 26 |
| Surface Condition ⁽²⁾ | 27 |
| Paint Chips ⁽³⁾ | 28 |

KEY: (Enter Code)

⁽¹⁾ V = Vinyl, M = Metal

W = Sealed Wood

U = Unsealed Wood

⁽²⁾ S = Smooth, R = Rough(Intact)

D = Rough(deteriorated)

⁽³⁾ 0 = No or 1 = Yes

Place
Bar Code
Here

Depth Wiped (inches)

Width Wiped (inches)

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other

DESCRIBE:

SURFACE TYPE

(Enter Code: S or W)

WIPE SAMPLE TYPE

(Enter Code: E, R, L, or P)

(Enter Window Number to be Sampled)

| | |
|----------------------------------|----|
| Window No.: | 24 |
| Surface Material ⁽¹⁾ | 26 |
| Surface Condition ⁽²⁾ | 27 |
| Paint Chips ⁽³⁾ | 28 |

KEY: (Enter Code)

⁽¹⁾ V, M, W, or U

⁽²⁾ S, R, or D

⁽³⁾ 0 or 1

Place
Bar Code
Here

Depth Wiped (inches)

Width Wiped (inches)

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other

DESCRIBE:

Supervisor: _____ Date: _____

11/23/92



Kennedy Krieger Institute Repair and Maintenance Study
WIPE DUST COLLECTION FORM FOR FIELD QC:
Field Blanks and Field Duplicates

DWELLING ID - ROUND

(Round: IN PI 02 06 12 18 24)

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|----------------------|----------------------|----------------------|---|----------------------|----------------------|
| <input type="text"/> | <input type="text"/> | <input type="text"/> | - | <input type="text"/> | <input type="text"/> |
|----------------------|----------------------|----------------------|---|----------------------|----------------------|

1

DATE

| | | | | | | | |
|----------------------|----------------------|---|----------------------|----------------------|---|----------------------|----------------------|
| <input type="text"/> | <input type="text"/> | - | <input type="text"/> | <input type="text"/> | - | <input type="text"/> | <input type="text"/> |
| m | m | | d | d | | y | y |

6

SAMPLER (Initials)

| | |
|----------------------|----------------------|
| <input type="text"/> | <input type="text"/> |
|----------------------|----------------------|

12

RECORDER (Initials)

| | |
|----------------------|----------------------|
| <input type="text"/> | <input type="text"/> |
|----------------------|----------------------|

14

WIPE FIELD BLANK SAMPLE ID
(One per dwelling)

Place FB
Bar Code
Here

16

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other
DESCRIBE:

| |
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| <input type="text"/> |
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22

WIPE FIELD DUPLICATE SAMPLE ID
(Not collected if sampling is done for Maryland
Department of the Environment)

Place FD
Bar Code
Here

23

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other
DESCRIBE:

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| <input type="text"/> |
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29

WIPE FIELD DUPLICATE LINK ID
(Put Bar Code of Dust Wipe Sample paired to Field Duplicate)

Place Bar Code
of DW Sample
Paired with FD

30

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other
DESCRIBE:

| |
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| <input type="text"/> |
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36

Supervisor: _____ Date: _____



Kennedy Krieger Institute Repair and Maintenance Study
WIPE DUST COLLECTION FORM
for Side by Side Wipe/Vac Mini Study

DWELLING ID - ROUND

(Round: IN PI 02 06 12 18 24)

DATE

SAMPLER (Initials)

RECORDER (Initials)

WIPE SAMPLE ID

Place
Bar Code
Here

SURFACE TYPE

F = Floor S = Window Sill W = Window Well

WINDOW PORTION WIPED

L = Left Half R = Right Half N = Not Applicable

Depth Wiped (inches)

Width Wiped (inches)

WIPE FIELD BLANK SAMPLE ID

(Collect only one per dwelling when side by side wipe/vacs are collected.)

Place Wipe FB
Bar Code
Here

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other
DESCRIBE:

WIPE FIELD DUPLICATE SAMPLE ID

(This must be a Floor Sample, collect only one per dwelling.)

Place Wipe FD
Bar Code
Here

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other
DESCRIBE:

DUST/VACUUM SAMPLE LINK

(Put Bar Code of Dust Vacuum Sample paired to the Wipe Sample)

Place Bar Code Here
of Dust Vacuum
Paired to the Wipe

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other
DESCRIBE:

Supervisor: _____ Date: _____

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Kennedy Krieger Institute Repair and Maintenance Study
SOIL CORE COLLECTION FORM

DWELLING ID - ROUND

(Round: IN PI 02 06 12 18 24)

| | | | | | |
|----------------------|----------------------|----------------------|---|----------------------|----------------------|
| <input type="text"/> | <input type="text"/> | <input type="text"/> | - | <input type="text"/> | <input type="text"/> |
|----------------------|----------------------|----------------------|---|----------------------|----------------------|

DATE

| | | | | | | | |
|----------------------|----------------------|---|----------------------|----------------------|---|----------------------|----------------------|
| <input type="text"/> | <input type="text"/> | - | <input type="text"/> | <input type="text"/> | - | <input type="text"/> | <input type="text"/> |
| m | m | | d | d | | y | y |

SAMPLER (Initials)

| | |
|----------------------|----------------------|
| <input type="text"/> | <input type="text"/> |
|----------------------|----------------------|

RECORDER (Initials)

| | |
|----------------------|----------------------|
| <input type="text"/> | <input type="text"/> |
|----------------------|----------------------|

SAMPLE ID

Place
Bar Code
Here

LOCATION

F = Front R = Rear S = Side

| |
|----------------------|
| <input type="text"/> |
|----------------------|

SURFACE TYPE

P = Property Line E = Entrance Way
D = Drip Line O = Other

| |
|----------------------|
| <input type="text"/> |
|----------------------|

SURFACE DESCRIPTION

G = Grass/Groundcover E = Exposed dirt
C = Combined

| |
|----------------------|
| <input type="text"/> |
|----------------------|

CHILD FLAG

Do any children in this household play in this area?

1 = Yes 0 = No 8 = Not Applicable 9 = Don't Know

| |
|----------------------|
| <input type="text"/> |
|----------------------|

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other
DESCRIBE:

| |
|----------------------|
| <input type="text"/> |
|----------------------|

FIELD BLANK SAMPLE ID

(One per dwelling when soil is collected.)

Place FB
Bar Code
Here

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other
DESCRIBE:

| |
|----------------------|
| <input type="text"/> |
|----------------------|

FIELD DUPLICATE SAMPLE ID

(If the last digit of the dwelling ID is a "6" then
collect a field duplicate, otherwise leave blank.)

Place FD
Bar Code
Here

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other
DESCRIBE:

| |
|----------------------|
| <input type="text"/> |
|----------------------|

DIAGRAM DEVIATIONS FROM SAMPLING PLAN ON BACK OF THIS FORM:

Supervisor: _____ Date: _____

11/16/92



**Kennedy Krieger Institute Repair and Maintenance Study
WATER SAMPLES COLLECTION FORM**

DWELLING ID - ROUND

(Round: IN PI 02 06 12 18 24)

| | | | | | | |
|----------------------|----------------------|----------------------|---|----------------------|----------------------|---|
| <input type="text"/> | <input type="text"/> | <input type="text"/> | - | <input type="text"/> | <input type="text"/> | 1 |
|----------------------|----------------------|----------------------|---|----------------------|----------------------|---|

DATE

| | | | | | | | | |
|----------------------|----------------------|---|----------------------|----------------------|---|----------------------|----------------------|---|
| <input type="text"/> | <input type="text"/> | - | <input type="text"/> | <input type="text"/> | - | <input type="text"/> | <input type="text"/> | 6 |
| m | m | | d | d | | y | y | |

SAMPLER (Initials)

| | | |
|----------------------|----------------------|----|
| <input type="text"/> | <input type="text"/> | 12 |
|----------------------|----------------------|----|

RECORDER (Initials)

| | | |
|----------------------|----------------------|----|
| <input type="text"/> | <input type="text"/> | 14 |
|----------------------|----------------------|----|

SAMPLE ID

| | |
|---------------------------|----|
| Place Bar Code Here | 16 |
|---------------------------|----|

LEVEL

0 = Basement 1 = 1st fl 2 = 2nd fl 3 = 3rd fl

| | |
|----------------------|----|
| <input type="text"/> | 22 |
|----------------------|----|

ROOM USAGE

KI = Kitchen BA = Bathroom OT = Other

| | | |
|----------------------|----------------------|----|
| <input type="text"/> | <input type="text"/> | 23 |
|----------------------|----------------------|----|

TIME SYSTEM FLUSH COMPLETED

(Use Military Time)

| | | | | |
|----------------------|----------------------|----------------------|----------------------|----|
| <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | 25 |
|----------------------|----------------------|----------------------|----------------------|----|

TIME SAMPLE COLLECTED

(Use Military Time)(Collect 2 hours after flush completed)

| | | | | |
|----------------------|----------------------|----------------------|----------------------|----|
| <input type="text"/> | <input type="text"/> | <input type="text"/> | <input type="text"/> | 29 |
|----------------------|----------------------|----------------------|----------------------|----|

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other

DESCRIBE:

| | |
|----------------------|----|
| <input type="text"/> | 33 |
|----------------------|----|

FIELD BLANK SAMPLE ID

(One per dwelling when water is collected.)

| | |
|------------------------------|----|
| Place FB Bar Code Here | 34 |
|------------------------------|----|

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other

DESCRIBE:

| | |
|----------------------|----|
| <input type="text"/> | 40 |
|----------------------|----|

FIELD DUPLICATE SAMPLE ID

(If the last digit of the dwelling ID is a "6" then collect a field duplicate, otherwise leave blank.)

| | |
|------------------------------|----|
| Place FD Bar Code Here | 41 |
|------------------------------|----|

COMMENT: 0 = No Comment (good sample) 1 = Sample Voided in Field 2 = Sample Collected with Protocol Deviation 3 = Other

DESCRIBE:

| | |
|----------------------|----|
| <input type="text"/> | 47 |
|----------------------|----|

Supervisor: _____ Date: _____

11/16/92



Kennedy Krieger Institute Repair and Maintenance Study
ENVIRONMENTAL SAMPLING CHECKLIST

DWELLING ID - ROUND

(Round: IN PI 02 06 12 18 24)

.

DATE

. .
 m m d d y y

| To Be Sampled | Sample Type | Location* | Completed |
|---------------|---------------------------------------|-----------|-----------|
| | <u>Vacuum/Dust</u> | | |
| | Floor Composites: | | |
| | 1st floor-- | | |
| | rooms w/windows | | |
| | 2nd floor-- | | |
| | rooms w/windows | | |
| | Across floors-- | | |
| | rooms wo/windows | | |
| | Window Composites - 1st Floor: | | |
| | all available sills | | |
| | all available wells | | |
| | Window Composites - 2nd Floor: | | |
| | all available sills | | |
| | all available wells | | |
| | Air/duct/upholstery | | |
| | Interior Entranceway | | |
| | Exterior Entranceway | | |
| | <u>Soil Cores</u> | | |
| | Near foundation | | |
| | Property boundary | | |
| | <u>Drinking Water</u> | | |
| | <u>Field QC</u> | | |
| | Vacuum/dust blank | | |
| | Vacuum/dust duplicate | | |
| | Soil blank | | |
| | Soil duplicate | | |
| | Water blank | | |
| | Water duplicate | | |
| | Wipe/dust blank | | |
| | Wipe/dust duplicate | | |
| | TOTAL SAMPLES | | |

*Indicate Room Letter or Window Number

Dwelling #320
2708 Laurel
Level: 1st Floor
Measurer: Marc Talle
Drawer: Carl Staples
Scale: 1"=4 ft





N ↗

Dwelling #320

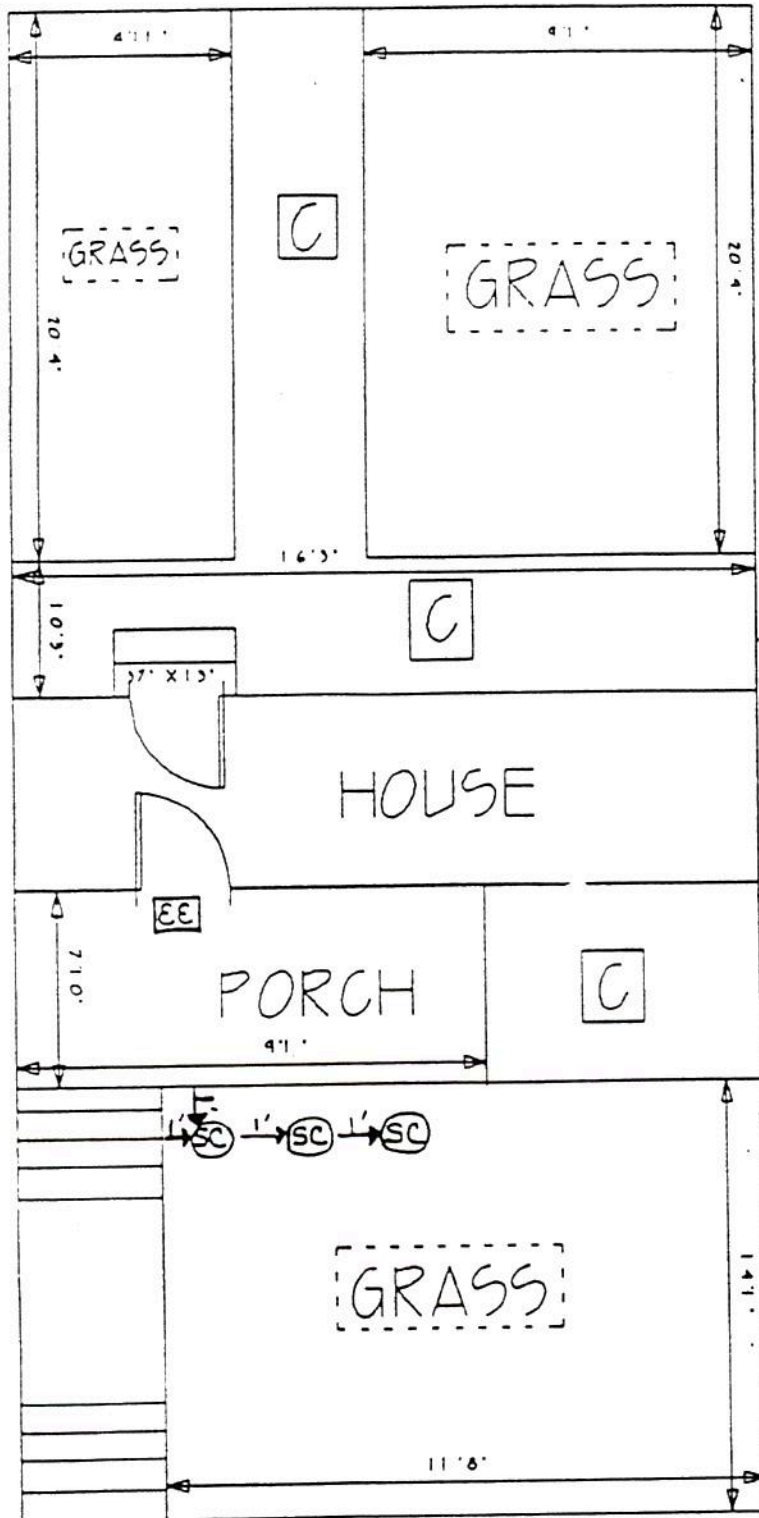
2708 Lauret

Level: Exterior

Measurer: Marc Talley

Drawer: Carl Staples

Scale: Not to Scale



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APPENDIX Q
BLOOD LEAD REPORTING FORM AND
ANODIC STRIPPING VOLTAMMETRY (ASV) ANALYSIS DATA SHEET

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**Kennedy Krieger Institute Repair and Maintenance Study
BLOOD COLLECTION AND REPORTING FORM**

I. COLLECTION

A. Child ID: (Dwelling ID, Child ID)

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B. Collection Round:
(IN PI 02 06 12 18 24)

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C. Collection Date:
Phlebotomist Initials: _____

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m m d d y y

Comments: (Code and Describe Below) 0 = No Comment(good sample); 1 = Sample Voided; 2 = Sample Collected with Protocol Deviation; 3 = Other
Describe:

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II. RESULT ASV

A. Analysis Date ASV PbB:
Analyst Initials: _____

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ASV PbB Values: ($\mu\text{g/dL}$)

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Comments: (Code and Describe Below) 0 = No Comment(good sample); 1 = Sample Voided; 2 = Sample Collected with Protocol Deviation; 3 = Other
Describe:

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B. Analysis Date FEP/HCT:
Analyst Initials: _____

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FEP: ($\mu\text{g/dL RBC}$)

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HCT: (%)

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Comments: (Code and Describe Below) 0 = No Comment(good sample); 1 = Sample Voided; 2 = Sample Collected with Protocol Deviation; 3 = Other
Describe:

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III. RESULTS: GFAA

A. Analysis Date GFAA PbB:
Analyst Initials: _____

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m m d d y y

GFAA PbB Values: ($\mu\text{g/dL}$)

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Comments: (Code and Describe Below) 0 = No Comment(good sample); 1 = Sample Voided; 2 = Sample Collected with Protocol Deviation; 3 = Other
Describe:

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DATE _____

MACHINE _____
TOWER _____
ELECTRODE _____
REAGENT _____

```
INITIAL POTENTIAL -1.090 v
FINAL POTENTIAL -0.150 v
SWEEP RATE 14.00 mv/step
REC. SET POINT -0.700 v
INT. SET POINT -0.500 v
```

STANDARDS 12/88
MATRIX Blood
ANALYSIS TIME 1 min
ANALYST

[illegible]

APPENDIX R

**PROTOCOLS FOR
PREPARATION OF DUST, SOIL AND WATER SAMPLES FOR TOTAL
LEAD ANALYSIS USING ACID DIGESTION:**

**PROTOCOL FOR THE DIGESTION OF
DUST, SOIL AND WATER SAMPLES USING MICROWAVE DIGESTION**

**MODIFIED METHOD 3050 FOR PREPARATION OF
VACUUM CASSETTE DUST SAMPLES USING HOTPLATE DIGESTION
(Backup Method)**

**MODIFIED METHOD 3050 FOR PREPARATION
OF SOIL SAMPLES USING HOTPLATE DIGESTION
(Backup Method)**

**MODIFIED METHOD 3020 FOR PREPARATION OF
DRINKING WATER SAMPLES USING HOTPLATE DIGESTION
(Backup Method)**

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Changes made to this protocol;

1. New Microwave Oven, MDS 2100, which has fiber optic temperature monitoring has replaced the MDS 2000. The text has been altered to reflect this.
2. Vacuum cassettes have been removed from the procedure and replaced by microwave digestion liners (Teflon[®] liners).
3. Quality control samples have been altered to reflect the changes in the number of samples. One SRM will now be used per batch. The SRM and method blank will be placed in one batch and the spike and spike duplicate will be placed in the second batch.

**PROTOCOL FOR THE DIGESTION OF
DUST, SOIL AND WATER SAMPLES USING MICROWAVE DIGESTION**

1.0 INTRODUCTION

- 1.1 Microwave digestion is proposed as a means of increasing throughput of samples and minimizing sample contamination relative to hotplate digestion without decreasing the efficiency of digestion.¹ The microwave digestion programs for dust, soil, and water (hereafter referred to as EPAD, EPAS, and EPAW) are modeled after existing Contract Laboratory Protocol (CLP) methods and proposed EPA microwave digestion protocols SW-846 Methods 3015 and 3051 for soil and water (see attached EPA microwave digestion protocols).² These microwave procedures are designed to replace the following appendices which were used in the Pilot Study: Appendix A (Modified Cincinnati Method), Appendix C (Modified Method 3050), Appendix D (Modified Method 3020). Hotplate methods will be used as backup methods of preparation of dust and soil (modified Method 3050), and water samples (modified Method 3020).

2.0 CLEANING OF GLASSWARE/TEFLON

- 2.1 All glassware and teflon will be soaked in Acationox detergent according to Appendix X: Protocol for Glassware/Plasticware Preparation (R&M QAPjP, Revision No. 3).
- 2.2 After washing, the glassware and teflon will be soaked in 10% HNO_3 (made with ASTM type I water) overnight.
- 2.3 The glassware and teflon will be rinsed seven times in ASTM type I water.
- 2.4 The glassware and teflon will be allowed to air dry and stored after drying until required.
- 2.5 The microwave can be used to clean the digestion liners as an alternative to 2.2 above. This is done by adding 10 mL of 1:1 HNO_3 to each digestion liner and placing the open digestion liner in the microwave. A quick heating program (10 minutes at 80 percent power) is used to clean the digestion liners. Steps 2.1 will be carried out before step 2.5.

3.0 SUMMARY OF METHOD

Note: Each sample preparation method is based on an expected total volume of digestion solution. Any shortfall in sample number must be made up by adding extra vessels to make up the total volume. For example, the Dust Sample Preparation (3.1) is based on 12 vessels, each containing 10 mL of 1:1 $\text{HNO}_3:\text{H}_2\text{O}$. If only 10 samples including QC are prepared for digestion then the technician must add 10 mL of digestion solution to the two extra vessels to make up the volume. Under no circumstances must a microwave program be run with less than the recommended number of vessels. Also, samples matrices should only be run using the recommended program for each matrix. For example, dust samples should not be run with water or soil samples.

3.1 Dust Sample Preparation

Dust collected by cyclone sampler in pretared 100 mL Teflon[®] liners is oven dried at 110°C, cooled, weighed and transferred to the microwave oven for digestion. The samples are digested, at pressure, in HNO_3 (1:1 with ASTM type I H_2O). The samples are heated at a wattage that will bring them to 160°C $\pm 4^\circ\text{C}$ in ten minutes and permits a slow rise to 165-170° during the second ten minutes. The digestate is filtered to remove insoluble material and the final dilution volume, made up with ASTM type I water, is 50 mL.

Note: This procedure is for 12 digestion vessels each containing 10mL 50% HNO_3 and must not be used with less than this number. Sample shortfall can be made up by adding extra reagent blanks to the turntable.

3.2 Soil Sample Preparation

The soil is dried at 110 °C, ground, and sieved (US#4, #10 sieve). A representative 0.5g \pm 0.020 g (weigh out precision) is transferred to a 120 mL Teflon[®] digestion liner and digested in 10 mL of 1:1 HNO_3 (made with ASTM type I H_2O). The samples are heated at a wattage that will bring them to 175°C $\pm 4^\circ\text{C}$ in 5.5 minutes and permits a slow rise to 175-180°C for the remaining 4.5 minutes. The digestate is then filtered to remove insoluble material and the final dilution volume, made up with ASTM type I water, is 50 mL.

Note: This procedure is for 12 digestion vessels each containing 10mL 50% HNO_3 and must not be used with less than this number. Sample shortfall can be made up by adding extra reagent blanks to the turntable.

3.3 Water Sample Preparation

A representative 45 mL of water sample is transferred to a 100 mL digestion liner and 5 mL of HNO_3 (concentrated) is added. The samples are heated at a wattage that will bring them to $160^\circ\text{C} \pm 4^\circ\text{C}$ in ten minutes and permits a slow rise to $165\text{--}170^\circ\text{C}$ during the second ten minutes. The digestate is then filtered to remove insoluble material and the final dilution volume is made up to 50 mL using ASTM type I water.

Note: This procedure is for 12 digestion vessels each containing 50 mL of solution (sample plus acid) and must not be used with less than this number. Sample shortfall can be made up by adding extra reagent blanks to the turntable.

4.0 APPARATUS AND MATERIALS

- 4.1 Microwave Oven: CEM Corporation (CEM) Microwave Digestion System 2100 (see Attachment 1 and Attachment 2 at the end of this document).
- 4.2 Teflon[®] PFA lined digestion liners (n=12), one of which will be connected to the pressure sensing and temperature monitoring vessel in order to profile the conditions of digestion (see Attachment 3). A graph of temperature and pressure with time will be printed at the end of digestion. The digestion liners are made from Teflon[®] PFA and have a 100 mL capacity. The digestion liners are capable of withstanding pressures up to 200 psi and temperatures up to 250°C .
- 4.3 Drying oven; VWR 1305U, $110^\circ \pm 5^\circ\text{C}$.
- 4.4 Rupture membranes for liner caps.
- 4.5 Balance; Mettler AM100, accurate to 0.001g, with a capacity of 100 g.
- 4.6 Class A volumetric glassware, 50 mL capacity.
- 4.7 Thermometer; accurate to 0.1°C .
- 4.8 Filter funnels; polypropylene.
- 4.9 Filter paper; Whatmann No. 41 or equivalent.
- 4.10 Nalgene sample bottles; 60 mL capacity, high density polyethylene (HDPE).

5.0 REAGENTS

- 5.1 HNO₃ (Nitric acid), 70-71%, Baker analyzed.
- 5.2 ASTM type I water.
- 5.3 Buffalo River Sediment (SRM#2704) with a certified lead level of 161 ± 17 µg/g.
- 5.4 Trace Elements in Water (SRM#1643c) (35.3 ± 0.9 ng Pb/mL).

6.0 MICROWAVE CALIBRATION

- 6.1 In microwave digestion the heat is provided by radiation to the samples which are contained in sealed pressure resistant digestion liners. The microwave energy is transformed into heat in the sample. When the digestion liner is pressurized by vaporization of the sample, even higher temperatures can be obtained than those at atmospheric pressure resulting in much reduced digestion times without the risk of sample loss from boiling. Because microwave ovens differ in their capacity to produce heat, each unit must be calibrated initially to ensure that the percent (%) power setting is providing the expected amount of watts and hence the required amount of heating to the sample. This is done by calibrating the microwave oven using the measured temperature rise in a liter of water exposed to a set amount of microwave heating. The resulting plot of different power settings against watts (calculated from the rise in temperature) is referred to as microwave calibration (see Attachments 4 and 5) and is used to transfer methods from one instrument to another and to monitor the performance of the instrument over time. See section 6.4 for frequency of calibration. More recent models, such as the MDS2100 that is used here, have temperature and pressure monitoring devices which can be used to measure and control the rate of heating, thereby eliminating the need to indirectly predict the required operating conditions.

In contrast to microwave procedures, hotplate digestion temperature is monitored by a thermometer in a flask of deionized water which is placed on the heating plate. The hotplate dial is adjusted by the digestion technician until the temperature of the water as read by the thermometer is 80-100°C. This temperature ensures that the samples will not boil, and thus splatter, causing unwanted losses.

- 6.2 Calibrate the microwave oven using the procedure described in Attachment 4 and 5.
- 6.3 A record of the calibration plot will be stored in the sample preparation laboratory. Attachment 5 shows a calibration plot of the microwave oven carried out at the Kennedy Krieger Institute.
- 6.4 Initially, the microwave will be calibrated every two weeks until the Laboratory Supervisor and QC Officer determined that less frequent calibration is acceptable based on charting of power output over time.
- 6.5 Determine the percent power which will give the required wattage.
- 6.6 A hard copy of the pressure and temperature versus time plot for each digestion will be kept on file in the sample preparation lab.

7.0 QUALITY CONTROL

- 7.1 Using the microwave oven, a digestion batch will be defined as 24 samples (including QC) prepared at the same time. Since the microwave turntable has a capacity of 12 samples, each digestion batch will be divided into two sub-batches of 12 samples.
- 7.2 For each 21 field samples prepared, 4 QC samples will be used to monitor the process, making a total of 24 samples. The QC will consist of; one method blank, one spike, one spike duplicate and one NIST Standard Reference Material (SRM). The spike and spike duplicate will be placed in one sub-batch while the method blank and SRM will be placed in the other sub-batch.
- 7.3 Method blanks will consist of adding the 10 mL of 1:1 HNO₃ (made with ASTM type I H₂O) to a clean digestion liner. Water sample method blanks will be prepared by adding 5 mL of concentrated HNO₃ to 45 mL of ASTM type I water. Method blanks will be carried through the digestion process with the other samples.
- 7.4 Spikes will consist of field samples spiked with a known amount of lead; 10 µg/mL Pb soil samples and 0.5 µg/mL Pb for

water samples. (Spikes will demonstrate, if there is a matrix interference due to the sample). Dust samples cannot be split into two for spiking. A 10.0 $\mu\text{g/mL}$ Pb reagent spike is prepared for dust samples by adding the digestion reagents to a quantity of spiking solution; 1 mL of 500 $\mu\text{g/mL}$ Pb is added to 9 mL of 1:1 HNO_3 in a 100 mL liner. After digestion, ASTM Type I water is used to make a final sample volume of 50 mL.

- 7.5 Spike duplicates, for soil and water samples will be prepared by splitting the sample to be spiked and spiking with the same amount of lead as in 7.4. These will provide a measure of the analyst's ability to duplicate the process. For dust samples a reagent spike will be used for the duplicate.
- 7.6 SRM samples will be prepared by weighing (or dispensing if the SRM is in solution) a known amount of SRM into a digestion liner and digesting in the same manner as the field samples.
- 7.7 Prepare the following two solutions using ASTM type I water as the diluent: a) 1:1 HNO_3 : H_2O (v/v). This will be used as the digestion solution for dust and soil samples. For the digestion of water samples we will use concentrated HNO_3 . b) Prepare a spike solution by adding 50 mL of 1000 $\mu\text{g/mL}$ Pb in 10% HNO_3 solution to a clean 100 mL volumetric bottle and making up to volume with 10% HNO_3 (made with ASTM type I water). This solution contains 500 $\mu\text{g/mL}$ Pb in 10% HNO_3 .

8.0 DIGESTION PROCEDURE FOR DUST SAMPLES

- 8.1 The dust samples will be received in 100 mL capped digestion liners. The digestion liners will be in barcoded ziplock bags which contain sheets of identical bar codes.
- 8.2 The digestion liners are dried in a drying oven at 110 °C overnight.
- 8.3 The digestion liners are reweighed and the weight of sample calculated. (See Appendix I for R&M QAPjP, Revision No.3)
- 8.4 For the method blank 10 mL of 1:1 HNO_3 (made with ASTM type I water) is added to the digestion liner. One digestion blank per digestion batch of 24 samples is prepared.
- 8.5 Since field dust samples cannot be split into two samples, spikes and spike duplicates will be prepared without using samples. The spikes and spike duplicates will be prepared by

adding 1 mL of 500 µg/L solution to 9 mL of 1:1 HNO₃ in clean digestion liners. The final concentration of the spike and spike duplicate, will be 10.0 µg/mL Pb in 50 mL.

- 8.6 Weigh 0.5g ± 0.020 g (weigh out precision) of SRM#2704 into a clean digestion liner and add 10 mL of 1:1 HNO₃ (made with H₂O).

Note: It is important to divide the QC samples between sub-batches in order to monitor the whole batch as one unit. To do this place the method blank and the SRM in one sub-batch and the spike and spike duplicate in the second sub-batch. The spike and spike duplicate should always be digested in the same sub-batch.

- 8.7 Place the digestion liners in the digestion liner vessels (see Attachment 6). Place a clean rupture membrane in each digestion vessel cap and cap the digestion vessels. Place the digestion vessels on the turntable in the microwave (see Attachment 3) and connect the digestion liner with the largest sample weight to the pressure monitor digestion liner. Recall the program EPAD for dust digestion and start the digestion. NOTE: The pressure monitor digestion vessel is a digestion vessel which is connected to a pressure transducer via a pressure sensing tube and acts as a monitor of the digestion process. If the pressure in the digestion liner rises above a preset maximum, then the microwave will stop heating until the pressure has reduced below the value. This is important when large samples liberate gases as by-products of digestion. We use 100 psi as the maximum value for pressure. This is prewritten into each program.
- 8.8 The total volume of sample is digested at the predetermined wattage which will bring the samples to 160 °C in ten minutes followed by a slow rise to 165-170 °C in 10 minutes.
- 8.9 Following completion of the program the samples are left to cool in the oven for 5 minutes.
- 8.10 Samples are filtered using Whatmann no 41 filter paper and the filtrate made to a final dilution volume, with ASTM type I water, of 50 mL.
- 8.11 The sample digestate is stored in high density barcoded polyethylene bottles (60 mL).

9.0 DIGESTION PROCEDURE FOR SOIL SAMPLES

- 9.1 Use soil which has been dried and homogenized according to Appendix T of the R&M QAPjP Document, Revision 2.
 - 9.2 Add a representative $0.5 \text{ g} \pm 0.020 \text{ g}$ (weigh out precision) of sample to a Teflon[®] PFA digestion liner.
 - 9.3 Add 10 mL of 1:1 HNO_3 to the digestion liner. If a vigorous reaction occurs, allow the reaction to stop before capping the digestion liner.
 - 9.4 Prepare a method blank by adding 10 mL of 1:1 HNO_3 to a clean digestion liner.
 - 9.5 Spiked soil sample and duplicate samples are prepared by weighing out two aliquots of ($0.5 \text{ g} \pm 0.020 \text{ g}$, weigh out precision) soil sample into clean, dry digestion liners. Add 9.0 mL of 1:1 HNO_3 and 1 mL of 500 $\mu\text{g/mL}$ Pb spiking solution to each liner. When the digestate is made up to 50 mL the final concentration of the spike is 10.0 $\mu\text{g/mL}$ Pb.
 - 9.6 Weigh out $0.5 \text{ g} \pm 0.020 \text{ g}$ of SRM#2704 into a clean digestion liner and add 10mL of 1:1 HNO_3 . This SRM will be placed in one sub-batch as a monitor of the digestion process for soils.
 - 9.7 Place the digestion liners in the digestion liner bodies. Place a clean rupture membrane in each cap and cap the digestion liners. Place the digestion liners in the turntable, using the highest weight for the pressure and temperature monitor digestion liner. Recall EPAS and start the digestion.
- NOTE: The pressure and temperature monitor digestion vessel is a digestion vessel which is connected to a pressure transducer via a pressure sensing tube and acts as a monitor of the digestion process. If the pressure in the digestion liner rises above a preset maximum, then the microwave will stop heating until the pressure has reduced below the value. We use 100 psi as the maximum value. Temperature is monitored by a optical fibre probe which is placed in a thermowell in the vessel.
- 9.8 The total sample volume will be heated at the predetermined wattage to bring the samples to 175 °C in 5.5 minutes and continue digestion at this temperature for the balance of the time period.

- 9.9 Allow the digestion liners to cool for 5 minutes before removing them from the turntable.
- 9.10 Make the sample up to 50 ml with ASTM type I water, in a volumetric bottle and filter. Add more water after filtration if necessary, to complete the makeup volume.
- 9.11 The sample digestate is stored in 60 ml high density polyethylene bottles.

10.0 DIGESTION PROCEDURE FOR WATER SAMPLES

- 10.1 Measure a 45 mL aliquot of the sample into a 120 mL Teflon[®]. PFA digestion liner using volumetric glassware.
- 10.2 Add 5 mL of HNO₃ to the digestion liner.
- 10.3 Record the weight of each digestion liner to ± 0.002 g.
- 10.4 Prepare a method blank by adding 5 mL of HNO₃ (concentrated) to 45 mL of ASTM type I water in a 100 mL digestion liner.
- 10.5 Water spikes and duplicates are prepared by adding 0.5 mL of spiking solution to 44.5 mL of field sample. Add 5.0 mL of concentrated HNO₃ to the digestion liner. The final concentration of the spike is 0.5 $\mu\text{g/mL}$.
- 10.6 Water SRM#1643c is prepared by adding 25 mL of the SRM to 20 mL of ASTM type I water and adding 5 mL of concentrated HNO₃. The final concentration of the solution will be 17.65 $\mu\text{g Pb/L}$ in 50 mL of solution.
- 10.7 The total volume of sample is heated at the predetermined wattage to bring the samples to 160 °C in 10 minutes and permit a slow rise to 165-170 °C during the second 10 minutes.
- 10.8 Allow the digestion liners to cool for 5 minutes.
- 10.9 Reweigh the digestion liners; if the losses are ≤ 0.5 g then continue with procedure; if losses are > 0.5 g then redigest the sample.
- 10.10 Make the samples up to 50 mL with ASTM type I water and filter. Add more water if necessary after filtration to complete the makeup volume.

11.0 ANALYSIS

- 11.1 Initial analysis of dust and soil samples will be carried out by ICP. Water samples will be analyzed by Graphite Furnace AA (GFAA). Samples found to be less than ten times the detection limit of the ICP will be analyzed by GFAA.
- 11.2 Standards will be made in 10% HNO₃, made with ASTM type I water.
- 11.3 The correlation coefficient for the standards will be > .995 otherwise recalibration will be necessary.
- 11.4 Immediately after calibration and before any samples are run, the lowest standard will be run 5 times in order to calculate the instrument detection limit (IDL).
- 11.5 The calibration will be checked for linearity with a low and high check solution. Initially these will be known as ICV and ICB and during the run as CCV and CCB.
- 11.6 The CCV, CCB pair will be run at a frequency of at least 10%, that is every 10 samples.
- 11.7 If the ICV or CCV are found to be not $\pm 10\%$ of their true value then the run will be considered out of control and the standard curve will be recalibrated.

12.0 RESULTS

- 12.1 Percent recovery of spikes are expected to fall within the $\pm 30\%$ range.
- 12.2 Percent recovery of SRM's are expected to fall within the $\pm 30\%$ range.
- 12.3 Report results on a lab bench sheet and attach to the printout of the raw data.

**MODIFIED METHOD 3050 FOR PREPARATION OF
TEFLON LINER DUST SAMPLES USING HOTPLATE DIGESTION**

1.0 SUMMARY OF METHOD

1.1 This analytical method is a modified version of Method 3050 from the SW-846, 3rd Edition manual. It is written in the manner which provides ease of execution relative to the published procedure. This method which employs hotplate methods will be used as a backup method to microwave digestion of cyclone dust samples. The primary focus of the modifications relative to the published procedure is a change in amounts of reagents used and a change in final dilution volume to accommodate the sample size. In addition, the method has been modified for the cyclone method of sampling. It should be noted that the procedure for preparing lead samples for analysis by GFAA does not use hydrochloric acid for digestion due to signal depression interferences by lead chloride. The final reflux is nitric acid (HNO_3) and the sample digestate will be approximately 10% (v/v) HNO_3 , following digestion.

1.2 The entire Teflon liner dust sample is digested in HNO_3 and hydrogen peroxide (H_2O_2). The digestates are diluted to final volume following a final reflux and cookdown using nitric acid.

2.0 APPARATUS AND MATERIALS

2.1 Beakers: Griffin 100-mL

2.2 Watch glasses

2.3 Forceps: polyethylene

2.4 Volumetric flasks with stoppers: 50-mL

2.5 Thermometers: red alcohol and covers range of 0 to 100C

2.6 Hot plates: capable of maintaining a temperature of 80 to 100°C

2.7 Centrifuge

2.8 Centrifuge tubes: polyethylene with screw caps, 50-mL capacity

2.9 Kimwipes

2.10 Screwdriver

3.0 REAGENTS

- 3.1 ASTM type I water: Minimum resistance of 16.67 megaohm-cm, or equivalent
- 3.2 Concentrated (70 to 71%) HNO_3 : Baker instra-analyzed, or equivalent
- 3.3 Hydrogen peroxide (30%), reagent grade

4.0 QUALITY CONTROL

- 4.1 For each group of samples processed, method blanks (Type I water and reagents) should be carried throughout the entire sample preparation and analytical process. Method blanks are processed at a frequency of 1 for every 20 actual field samples. A minimum of 1 method blank is required for each batch processed.
- 4.2 Spiked samples are processed on a routine basis to determine an estimate of method accuracy expressed as percent recovery, relative to the true spiked value. A spiked sample is a sample aliquot (split from an original sample) which is spiked with a known amount of analyte. These samples are performed to provide accuracy data on the sample batch processing. The work assignment leader will provide direction on the analytes and spiking level to be used for the specific project. Spiked samples are processed at a frequency of 1 for every 20 actual field samples. A minimum of 1 spike is required for each batch processed. Because the actual field samples cannot be split, spikes will be performed on reagent blanks as specified in Appendix R, Microwave Digestion Protocol, section 7.4.
- 4.3 Spike Duplicate samples will be prepared in a manner identical to that described in Section 4.2. These are prepared to provide precision data on sample batch processing. Duplicate spiked samples are processed at a frequency of 1 for every 20 actual field samples. A minimum of 1 spike duplicate is required for each batch processed. Because the actual field samples cannot be split, spikes will be performed on reagent blanks as opposed to an aliquot of a field sample.
- 4.4 As an additional estimate of method accuracy standard

reference materials (SRM) are also incorporated into each analytical batch in a blind fashion and are not identified to the analyst.

5.0 PROCEDURE

NOTES: All reagent sources (lot #s) used for sample preparation must be recorded in a laboratory notebook. In addition, any inadvertent deviations to this procedure, unusual happenings or observations must also be recorded on a real-time basis as samples are processed. The laboratory task leader must be informed in a manner which permits as close to real-time action as possible if any deviations or unusual happenings occur in order to take any needed corrective actions.

All samples in a processing batch must be treated equally. For example, if one sample requires additional H_2O_2 , then all samples in that batch must be given additional peroxide.

- 5.1 Care should be taken during the execution of each step of the following procedure to ensure the sample losses do not occur due to spillage and the gains do not occur due to contamination.
- 5.2 Each teflon liner should arrive at the sample preparation laboratory closed, undamaged, and sealed inside a barcode labeled plastic bag inside a gallon plastic bag with extra corresponding barcode labels to be used for laboratory forms and glassware labeling. Note any samples which do not meet this criterion.
- 5.3 Label 100-mL Griffin beakers for each Teflon liner dust sample and associated quality control sample to be processed.
- 5.4 The Teflon liner dust samples do not require weighing by the LAB. (weighing is performed by the gravimetrics lab prior to submission to the sample preparation laboratory). Transfer each entire teflon liner dust sample (use blank teflon liners for spikes and spike duplicates) into labelled Griffin beakers using the following procedure.
 - 5.4.1 Carefully pry open and remove the top section of the teflon liner sample using a clean flat-edged

screw driver, or equivalent tool (see Figure A-1). Place the top section upside down (in a manner which will not cause sample loss) on a clean Kimwipe.

- 5.4.2 Gently empty the loose dust from the liner into a labeled beaker. Care should be taken to prevent sample losses due to blowing or scattering of the dust.
 - 5.4.3 Carefully rinse the inside of both the top section and remaining teflon liner with 5 to 10 mL of 10% (v/v) HNO_3 , transferring the rinse solution to the digestion beaker.
- 5.5 For spikes and spike duplicates add the appropriate amount of analyte into the appropriate beakers containing the sample aliquots chosen for spiking.
- 5.6 Perform digestion of each sample as follows.
- 5.6.1. Add 5 mL of 1:1 HNO_3 to each beaker, gently swirl to mix, and cover with a watch glass. Gently heat the sample to 85 to 100C and reflux for 10 to 15 min without boiling. (Temperature is continuously monitored by having on the hot plate a thermometer inside a beaker or flask containing small volume of water.) Allow the sample to cool, add 2.5 mL of concentrated HNO_3 , replace the watch glass and reflux for 30 min without boiling. Repeat this last step to ensure complete oxidation. Allow the solution to evaporate to approximately 5 mL without boiling, while maintaining a covering of solution over the bottom of the beaker. Note: Because ribbed watch glasses are not available, the watch glasses may be removed to accelerate volume reduction while assuring minimal contamination of the samples through careful observation. When removing these watch glasses, care must be exercised to avoid losses by rinsing them with a minimum amount of Type I water (rinsed into the sample beaker) and avoiding contaminating them by placing them upside down on new clean KIMWIPES. There should be minimum activity in the hood area and increased separation between beakers during this step.

- 5.6.2 After Step 5.6 has been completed and the sample has cooled, add 2 mL Type I water and 2 mL of 30% hydrogen peroxide (H_2O_2). Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.
- 5.6.3 Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Note: Do not add more than a total of 5 mL of 30% H_2O_2 even if effervescence has not been reduced to a minimal level.
- 5.6.4 Continue heating the acid-peroxide digestate carefully until the volume has been reduced to approximately 2.5 mL. Note: The watch glass covers may also be removed during this step using the same precautions as noted in Step 5.6. Care should be taken to ensure that the samples do not cook to dryness.
- 5.6.5 Allow the digestates to cool, rinse the beaker walls and bottom of the watch glass into the digestate, and quantitatively transfer to a 25 mL volumetric flask. Dilute to volume with Type I water.
- 5.7 Particulates in the digestate should then be removed by filtration, by centrifugation, or by allowing the sample to settle prior to instrumental measurement.
- 5.8 The diluted digestate solution contains approximately 10% (v/v) HNO_3 . Calibration standards used for instrumental measurement should be made with this concentration of HNO_3 .

**MODIFIED METHOD 3050 FOR PREPARATION
OF SOIL SAMPLES USING HOTPLATE DIGESTION**

1.0 SUMMARY OF METHOD

- 1.1 This method is a modified version of Method 3050 from the SW-846, 3rd Edition manual. It is written in a manner which provides ease of execution relative to the published procedure. This method which employs hotplate methods will be used as a backup method to microwave digestion of soil samples. This method is used for the drying, homogenization, and acid digestion of soil samples and associated quality control (QC) samples for ICP-AES analysis of Pb. The primary focus of the modifications relative to the published procedure is to include a sample drying and homogenization procedure to improve method accuracy and precision for the soil sample matrix, and a change in final dilution volume accommodate the sample size. It should be noted that the modified procedure for preparing soil samples does not use hydrochloric acid for digestion. The final reflux is nitric acid (HNO_3) and the sample digestate will be approximately 10% (v/v) HNO_3 following digestion.
- 1.2 A drying and homogenization technique is required in order to remove moisture and create a more representative sample with which to sub-sample for digestion. The procedure includes drying and sieving each sample, grinding and resieving dry material for an appropriate mixture grain size for the 3050 digestion. All soil samples received will be processed in an identical manner including the blind reference material.
- 1.3 The dried and homogenized soil samples from the field, and associated QC samples are digested in HNO_3 and hydrogen peroxide (H_2O_2). The digestates are diluted to final volume following a final reflux and cookdown using nitric acid.

2.0 APPARATUS AND MATERIALS

- 2.1 US. standard sieve #4 (4.7 mm): plastic or stainless steel
- 2.2 US. standard sieve #10 (1.9 mm): plastic or stainless steel
- 2.3 Mortar and pestle: porcelain
- 2.4 Alternate grinding apparatus: shatter boxes or mixer mill

- 2.5 Drying oven capable of maintaining a temperature of 100-120°C
- 2.6 Beakers: Griffin 100-mL
- 2.7 Watch glasses
- 2.8 Volumetric flasks with stoppers: 50-mL
- 2.9 Thermometers: red alcohol and covers range of 0-110°C
- 2.10 Hot plates: capable of maintaining a temperature of 80 to 100°C
- 2.11 Centrifuge
- 2.12 Centrifuge tubes: polyethylene with screw caps, 50-mL capacity
- 2.13 Kimwipes
- 2.10 Screwdriver

Note: Stainless steel or plastic sleeves must be used instead of the standard brass sleeves to alleviate possible Pb contamination of the soil samples from contact with solder of the brass sieves.

3.0 REAGENTS

- 3.1 ASTM type I water: Minimum resistance of 16.67 megaohm-cm, or equivalent
- 3.2 Concentrated (70 to 71%) HNO₃: Baker intra-analyzed, or equivalent
- 3.3 Hydrogen peroxide (30%)
- 3.4 Acetone: high purity reagent grade

4.0 QUALITY CONTROL

- 4.1 For each group of samples processed, method (digestion) blanks (Type I water and reagents) should be carried throughout the entire sample preparation and analytical process. The blanks will be useful in determining if the samples are being contaminated. Method blanks are processed at a frequency of 1 for every 20 actual field samples. A minimum of 1 method

blank is required for each batch processed.

- 4.2 Spiked samples are processed on a routine basis to determine an estimate of method accuracy expressed as percent recovery, relative to the true spiked value. A spiked sample is a sample aliquot (split from an original sample) which is spiked with a known amount of analyte. These samples are performed to provide accuracy data on the sample batch processing. The work project leader will provide direction on the analytes and spiking level to be used for the specific project. Spiked samples are processed at a frequency of 1 for every 20 actual field samples. A minimum of 1 spike is required for each batch processed.
- 4.3 Spike Duplicate samples will be prepared in a manner identical to that described in Section 4.2. These are prepared to provide precision data on sample batch processing. Duplicate spiked samples are processed at a frequency of 1 for every 20 actual field samples. A minimum of 1 spike duplicate is required for each batch processed.
- 4.4 As an additional estimate of method accuracy standard reference materials (SRM) are also incorporated into each analytical batch in a blind fashion and are not identified to the analyst.

5.0 PROCEDURE

NOTES: All reagent sources (lot #s) used for sample preparation must be recorded in a laboratory notebook. In addition, any inadvertent deviations to this procedure, unusual happenings or observations must also be recorded on a real-time basis as samples are processed. The laboratory task leader must be informed in a manner which permits as close to real-time action as possible if any deviations or unusual happenings occur in order to take any needed corrective actions.

All samples in a processing batch must be treated equally. For example, if one sample requires additional hydrogen peroxide, then all samples in that batch must be given additional peroxide.

- 5.1 Each sample received from the field should be sealed in a labeled one quart ziplock plastic bag with an extra one quart ziplock plastic bag inside a gallon plastic bag that contain extra corresponding barcode labels to be used for laboratory

forms and glassware labeling. Note any samples which do not meet these criteria.

5.2 Perform the drying and homogenization steps for each soil sample as listed below.

- 5.2.1 If possible, break up each of the soil cores within the ziplock bags prior to removal. Place on a clean US standard #4 (4.7 mm) sieve to remove any coarse grains and/or root material. Discard material retained on the sieve. Clean sieve between samples by tapping or using forced air or other dry method to prevent cross-contamination. (This must be done in a location well removed from the samples in process.)
Note: This step may be deleted if soils are notably finer than the #4 (4.7 mm) sieve.
- 5.2.2 Label an acid cleaned 100 mL Griffin Beaker with a high temperature wax pen. Transfer sieved portion to the labeled 100 mL Griffin beaker with watch glass and place in the drying oven overnight or for a minimum of 8 hours at a temperature of 110 ± 10 °C.
- 5.2.3 Remove the beakers containing the samples using tongs and allow them to cool to room temperature.
- 5.2.4 Grind each sample using a porcelain mortar and pestle, or other appropriate homogenization apparatus such as a shatterbox or mixer well.
- 5.2.5 Rinse the grinding apparatus with Type I water and follow with an acetone rinse for drying in order to prevent cross contamination between samples.
- 5.2.6 Place homogenized sample on a US standard #10 (1.9 mm) stainless steel or plastic sieve and swirl. Discard retained material. Clean sieve between samples as stated in Step 5.2.
- 5.2.7 Transfer sieved portion to a labeled Griffin beaker from Step 5.3 and place in drying oven for 24 hours at 110 ± 10 °C. Remove from oven, place in a desiccator and allow to cool to room temperature.

- 5.2.8 Store the dried, homogenized, and sieved soil samples in the supplied new quart ziplock bag and label it with a corresponding barcode label.
- 5.3 Label acid cleaned 100-mL Griffin beakers and watch glasses for performing the digestion of each soiled sample and associated QC sample. Perform digestion of each sample as listed below.
- 5.3.1 For each sample, weigh to the nearest 0.001 g and transfer to a labeled Griffin beaker a 0.5 ± 0.01 g portion of sample. Record the sample weights.
- 5.3.2 Add 5 mL of 1:1 HNO_3 to each beaker, gently swirl to mix, and cover with a watch glass. Gently heat the sample to 85 to 100 C and reflux for 10 to 15 min without boiling. (Temperature is continuously monitored by having on the hot plate a thermometer inside a beaker or flask containing small volume of water.) Allow the sample to cool, add 2.5 mL of concentrated HNO_3 , replace the watch glass and reflux for 30 min without boiling. Repeat this last step to ensure complete oxidation. Allow the solution to evaporate to 5 mL without boiling, while maintaining a covering of solution over the bottom of the beaker.
- Note: Because ribbed watch glasses are not available, the watch glasses may be removed to accelerate volume reduction while assuring minimal contamination of the samples through careful observation. When removing these watch glasses, care must be exercised to avoid losses by rinsing them with a minimum amount of Type I water (rinsed into the sample beaker) and avoiding contaminating them by placing them upside down on new clean KIMWIPES. There should be minimum activity in the hood area and increased separation between beakers during this step.
- 5.3.3 After Step 5.3.2 has been completed and the sample has cooled, add 2 mL Type I water and 2 mL of 30% hydrogen peroxide (H_2O_2). Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to

ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.

5.3.4 Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Note: Do not add more than a total of 5 mL of 30% H_2O_2 even if effervescence has not been reduced to a minimal level.

5.3.5 Continue heating the acid-peroxide digestate carefully until the volume has been reduced to approximately 5 mL.

Note: The watch glass covers may also be removed during this step using the same precautions as noted in Step 5.12. Care should be taken to ensure that the samples do not cook to dryness.

5.3.6 Allow the digestates to cool, rinse the beaker walls and bottom of the watch glass with Type I water and quantitatively transfer to a 50 mL volumetric flask. Dilute to volume with Type I water.

5.4 Particulates in the digestate should then be removed by filtration, by centrifugation, or by allowing the sample to settle prior to instrumental measurement.

5.5 The diluted digestate solution contains approximately 10% (v/v) HNO_3 . Calibration standards used for instrumental measurement should be made with this level of HNO_3 .

**MODIFIED METHOD 3020 FOR PREPARATION OF
DRINKING WATER SAMPLES USING HOTPLATE DIGESTION**

1.0 SUMMARY OF METHOD

- 1.1 This method is a modified version of Method 3020 from the SW-846, 3rd Edition manual. It is written in the manner which provides ease of execution relative to the published procedure. This method which employs hotplate methods will be used as a backup method to microwave digestion of drinking water samples. This method is used for the acid digestion of drinking water samples and associated quality control (QC) samples for GFAA analysis of Pb. The primary focus of the modifications relative to the published procedure is a reduce the sample size from 100-mL to 50-mL. It is noted that the reagent volumes and final dilution volumes for the digestion are reduced proportionally to the sample size reduction.

2.0 APPARATUS AND MATERIALS

- 2.1 Beakers: Griffin 100-mL
2.2 Watch glasses
2.3 Volumetric pipettes: glass, class A with 50-mL capacity
2.4 Volumetric flasks with stoppers: 50-mL
2.5 Thermometers: red alcohol and covers range of 0-100°C
2.6 Hot plates: capable of maintaining a temperature of 80-100°C
2.7 Centrifuge
2.8 Centrifuge tubes: polythylene with screw caps, 50-mL capacity
2.9 Kimwipes

3.0 REAGENTS

- 3.1 ASTM type I water: Minimum resistance of 16.67 megaohm-cm, or equivalent
3.2 Concentrated (70 to 71%) HNO₃: Baker instra-analyzed, or equivalent

4.0 QUALITY CONTROL

- 4.1 For each group of samples processed, method (digestion) blanks (Type I water and reagents) should be carried throughout the entire sample preparation and analytical process. Method blanks are processed at a frequency of 1 for every 20 actual field samples. A minimum of 1 method blank is required for each batch processed.

- 4.2 Spiked samples are processed on a routine basis to determine an estimate of method accuracy expressed as percent recovery, relative to the true spiked value. A spiked sample is a sample aliquot (split from an original sample) which is spiked with a known amount of analyte. These samples are performed to provide accuracy data on the sample batch processing. The Lab Supervisor will provide direction on the analytes and spiking level to be used for the specific project. Spiked samples are processed at a frequency of 1 for every 20 actual field samples. A minimum of 1 spike is required for each batch processed.
- 4.3 Spike Duplicate samples will be prepared in a manner identical to that described in Section 4.2. These are prepared to provide precision data on sample batch processing. Duplicate spiked samples are processed at a frequency of 1 for every 20 actual field samples. A minimum of 1 spike duplicate is required for each batch processed
- 4.4 As an additional estimate of method accuracy standard reference materials (SRM) are also incorporated into each analytical batch in a blind fashion and are not identified to the analyst.

5.0 PROCEDURE

NOTES: All reagent sources (lot #s) used for sample preparation must be recorded in a laboratory notebook. In addition, any inadvertent deviations to this procedure, unusual happenings or observations must also be recorded on a real-time basis as samples are processed. The laboratory task leader must be informed in a manner which permits as close to real-time action as possible if any deviations or unusual happenings occur in order to take any needed corrective actions.

All samples in a processing batch must be treated equally. For example, if one sample requires additional hydrogen peroxide, then all samples in that batch must be given additional peroxide.

- 5.1 Each sampled received from the field should be contained in a barcode labeled 500-mL plastic bottle inside a plastic ziplock gallon bag inside another plastic ziplock gallon bag with corresponding barcode labels. The lid should be sealed with electrical tape. Note any samples which do not meet this criterion.

5.2 Label acid cleaned 100-mL Griffin beakers and watch glasses for each drinking water sample and associate QC sample to be processed. Perform the following digestion procedure for each sample as described below.

5.2.1 For each digestion procedure, transfer a 50-mL representative aliquot of the well mixed sample to a 100-mL Griffin beaker. Add 1.5 mL of concentrated HNO_3 . Cover the beaker with a watch glass. Place the beaker on a hot plate maintained at 85 to 100°C and reflux for 15 to 20 minutes without boiling. (Temperature is continuously monitored by having on the hot plate a thermometer inside a beaker or flask containing small volume of water.) Cautiously evaporate each sample to a low volume (5 mL), making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry. Cool and add another 1.5-mL portion of concentrated HNO_3 . Cover the beakers with watch glasses and return to the hot plate. Allow the samples to gently reflux at 85 to 100°C for 15 to 20 minutes without boiling.

5.2.3 Continue heating, adding additional concentrated HNO_3 as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing). When the digestion is complete, evaporate to a low volume (5 mL) without boiling while maintaining a covering of solution over the bottom of the beaker. (Note: Because ribbed watch glasses are not available, the watch glasses may be removed to accelerate volume reduction while assuring minimal contamination of the samples through careful observation. When removing these watch glasses, care must be exercised to avoid losses by rinsing them with a minimum amount of Type I water (rinsed into the sample beaker) and avoiding contaminating them by placing them upside down on new clean KIMWIPES. There should be minimum activity in the hood area and increased separation between beakers during this step.) Remove the beaker from the hot plate and add approximately 5 mL of Type I water, mix, and continue warming the beaker for 10 to 15 min to allow additional solubilization of any residue to occur. Any residue remaining

after 15 min can be ignored and transferred as described in the next step.

- 5.2.3 Allow the digestates to cool, rinse the beaker walls and bottom of the watch glass with Type I water into the digestate, and quantitatively transfer to a 50 mL volumetric flask. Dilute to volume with Type I water.
- 5.7 Particulates in the digestate should then be removed by filtration, by centrifugation, or by allowing the sample to settle prior to instrumental measurement.
- 5.8 The diluted digestate solution contains approximately 10% (v/v) HNO_3 . Calibration standards used for instrumental measurement should be made with this level of HNO_3 .

INSTRUMENT DESCRIPTION

Operational Components

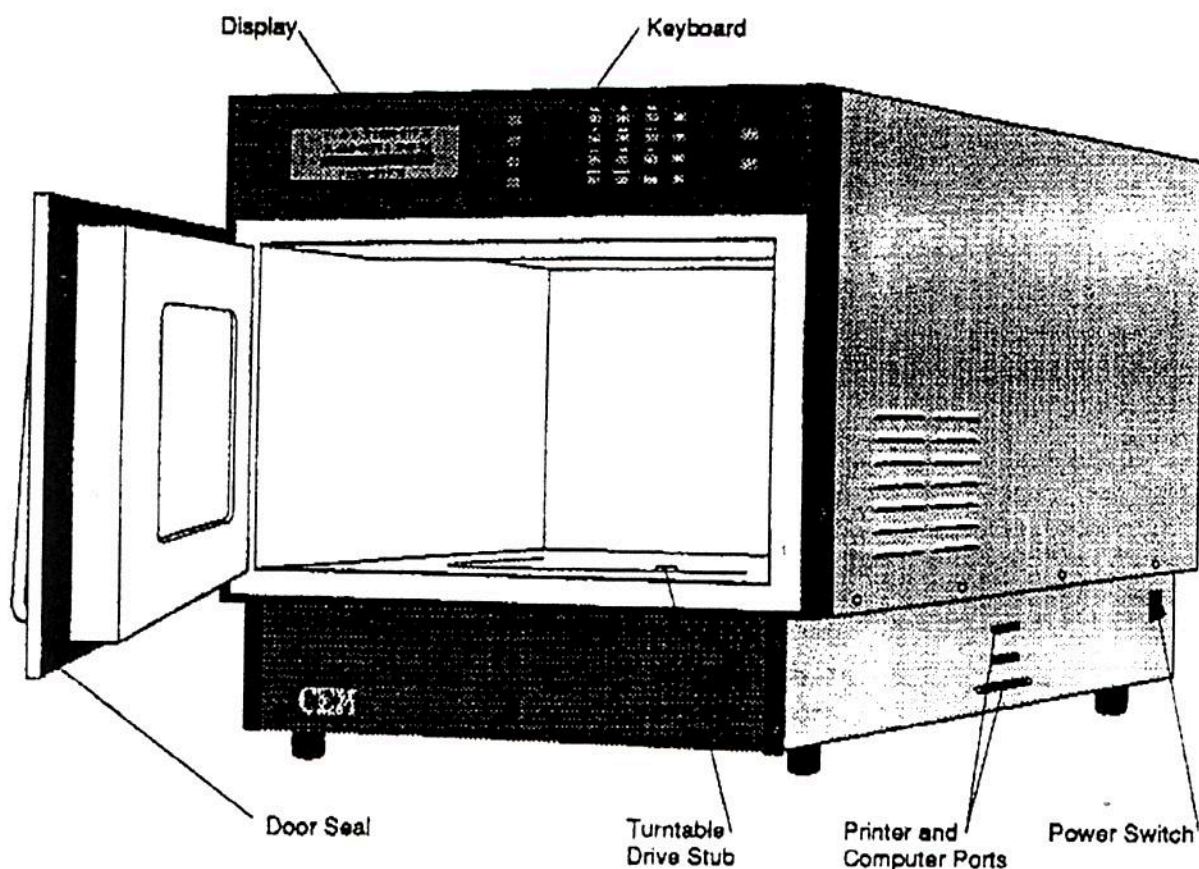


Figure 3. MDS-2100 Front View

- ☐ Display - shows menus, method parameters and instrument status on an 8 line x 40 character LCD display.
- ☐ Keyboard - allows the operator to control operational parameters such as power, time, pressure, and method names.
- ☐ Door Seal - ensures tight fit between door and interior cavity of the MDS-2100 to prevent microwave leakage.
- ☐ Turntable Drive Stub - allows the turntable drive shaft to pass through the cavity floor and engage the turntable.
- ☐ Printer and Computer Ports - allow communication with external devices for display and printout of data. See "System Software Setup" on page III-1 for detailed information.
- ☐ Power Switch - turns AC power to the instrument on and off.

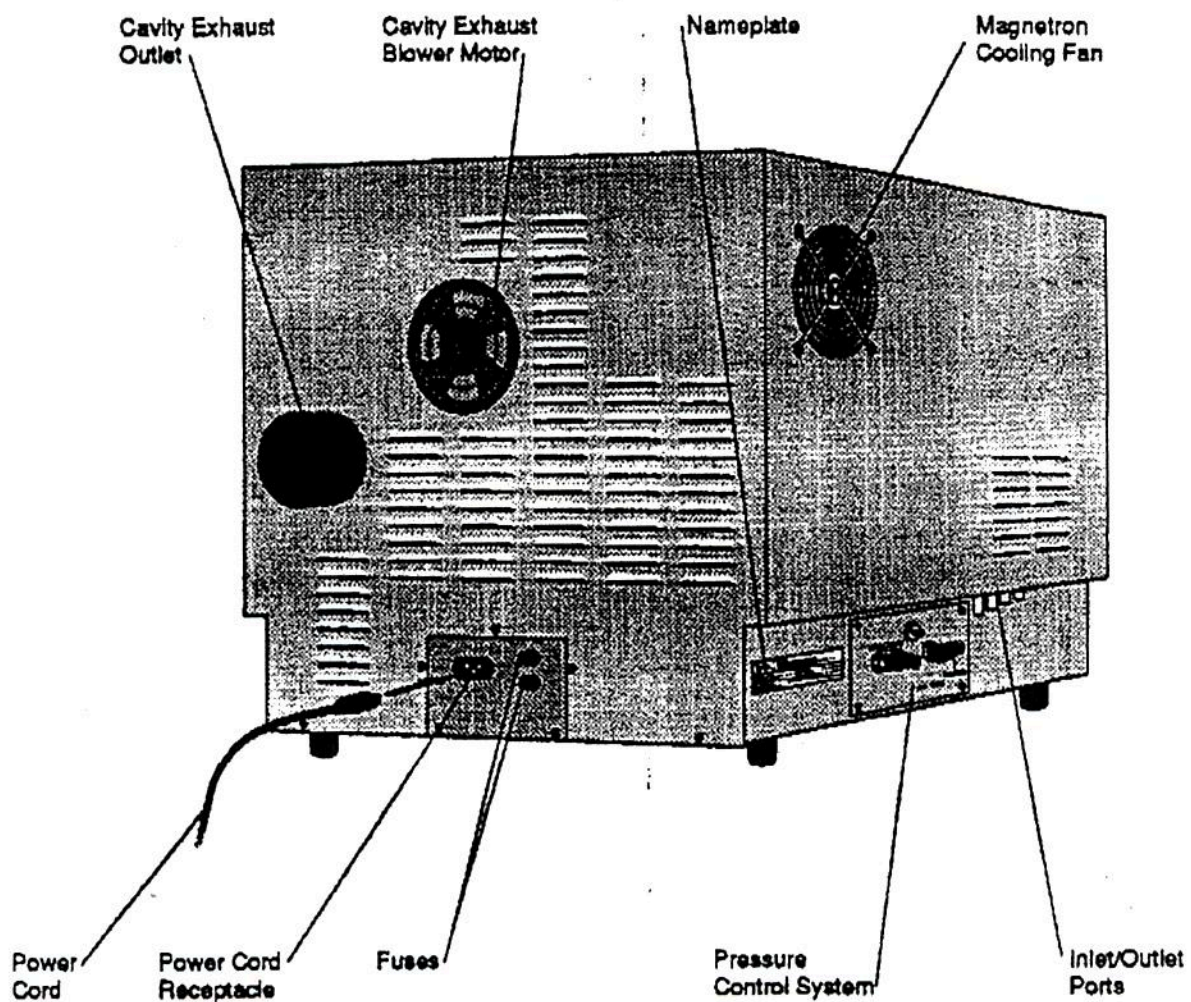


Figure 5. MDS-2100 Rear View

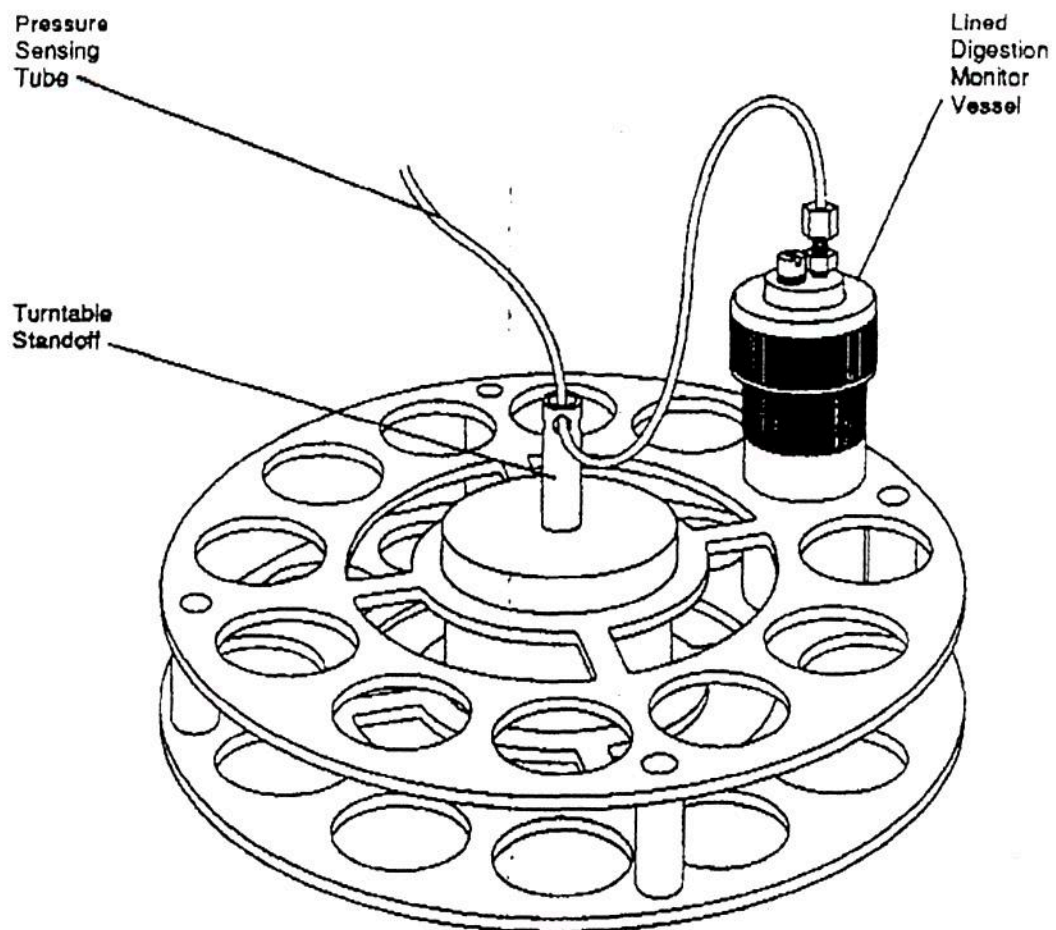


Figure 16. Routing of the Pressure Sensing Tube (Lined Digestion Vessel)

Microwave Power Measurement

The MDS-2100 has a microwave power output of approximately 950 watts at 100% power. Use the following procedure to determine power output.

1. Remove the turntable and drive lug from the microwave cavity.
2. Enter 5 minutes of time and 100% power in the Inorganic Sample (one stage) program. Program the fan speed for 0 percent and the pressure for 0 psi.
3. Press **START** and allow the instrument to operate for the programmed time to allow the magnetron and its power supply to warm up.
4. Reprogram Quick Digest with 100 percent power, 2 minutes time, 0 psi pressure, and 0 percent fan speed.
5. Place 1000 mL of room temperature deionized water in a 1000 mL Teflon® or polypropylene beaker.
6. Using a thermometer with 0.1 °C gradations, measure and record the initial water temperature, T_i .
7. Remove the thermometer from the beaker. Carefully place the beaker in the right front corner of the cavity. Gently close the door to avoid spilling any of the water.
8. Press **START**.
9. At the end of the 2-minute programmed time, remove the beaker from the microwave cavity. Stir the water thoroughly for 30 seconds, then measure and record the peak temperature reading. This is the final temperature, T_f .

The microwave power output is calculated as follows:

$$P \text{ (microwave power in W)} = (35 \text{ W/}^\circ\text{C}) (\Delta T)$$

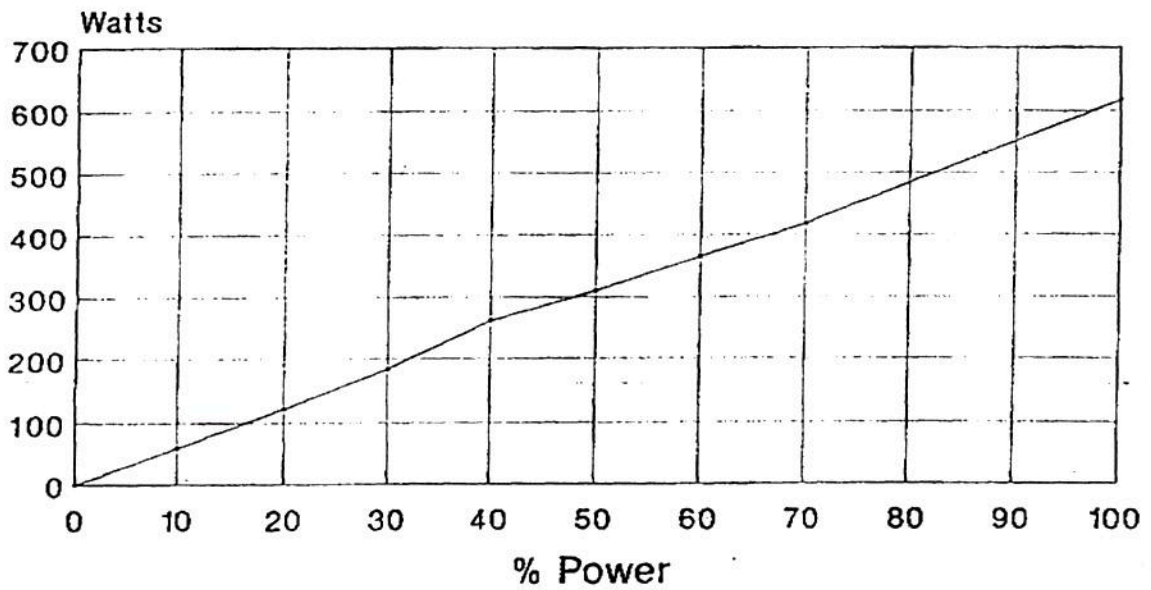
where ΔT , the change in water temperature, is $T_f - T_i$
 T_f = final temperature in °C
 T_i = initial temperature in °C

For example, if the initial water temperature (T_i) were 20.1 °C and the final water temperature (T_f) were 47.9 °C, then the power output would be

$$P = 35 \text{ W/}^\circ\text{C} (47.9^\circ\text{C} - 20.1^\circ\text{C}) = 973 \text{ W}$$

If the measured power is below 900 W, repeat the microwave power measurement. If the power remains less than 900 W, the instrument is not producing adequate microwave power. See "Troubleshooting Guide," page V-7.

% Power vs. Watts MSDS



Series 1

The formula used to calculate Watts is included in Attachment 6.

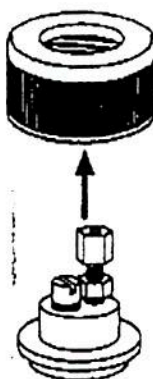


Figure 13. Pressure Control Cap and Cover Assembly for Lined Digestion Vessels

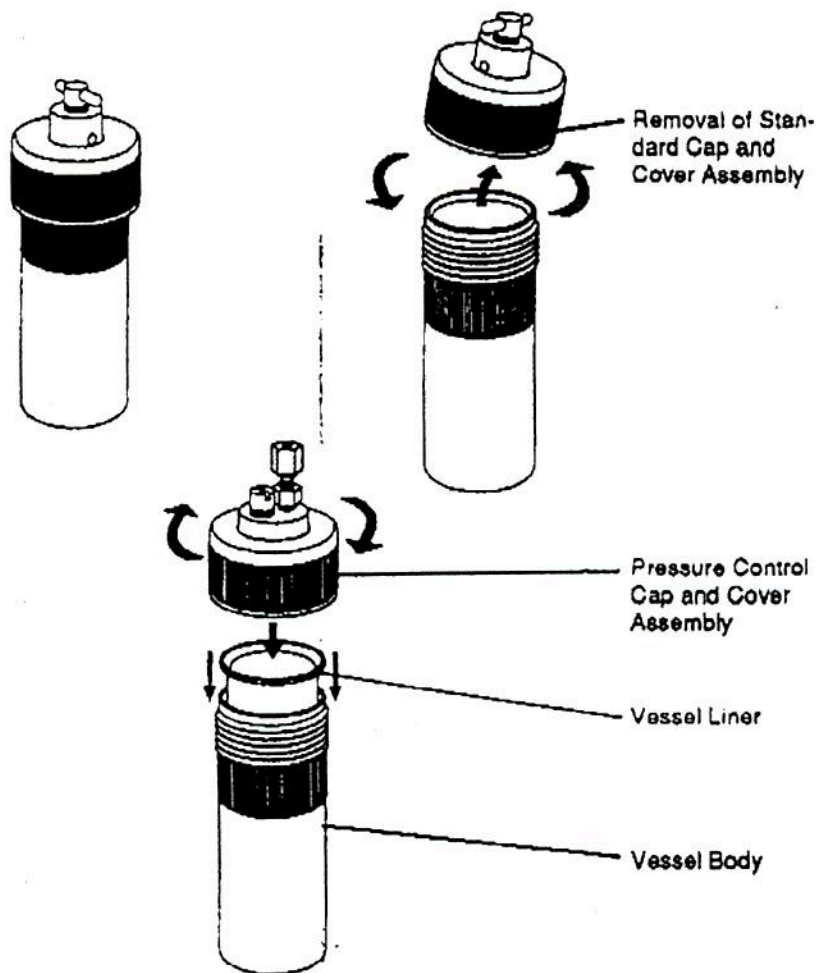


Figure 14. Installation of Pressure Control Cap and Cover Assembly on a Lined Digestion Vessel